

# The Journal *of the* SOCIETY OF DYERS AND COLOURISTS

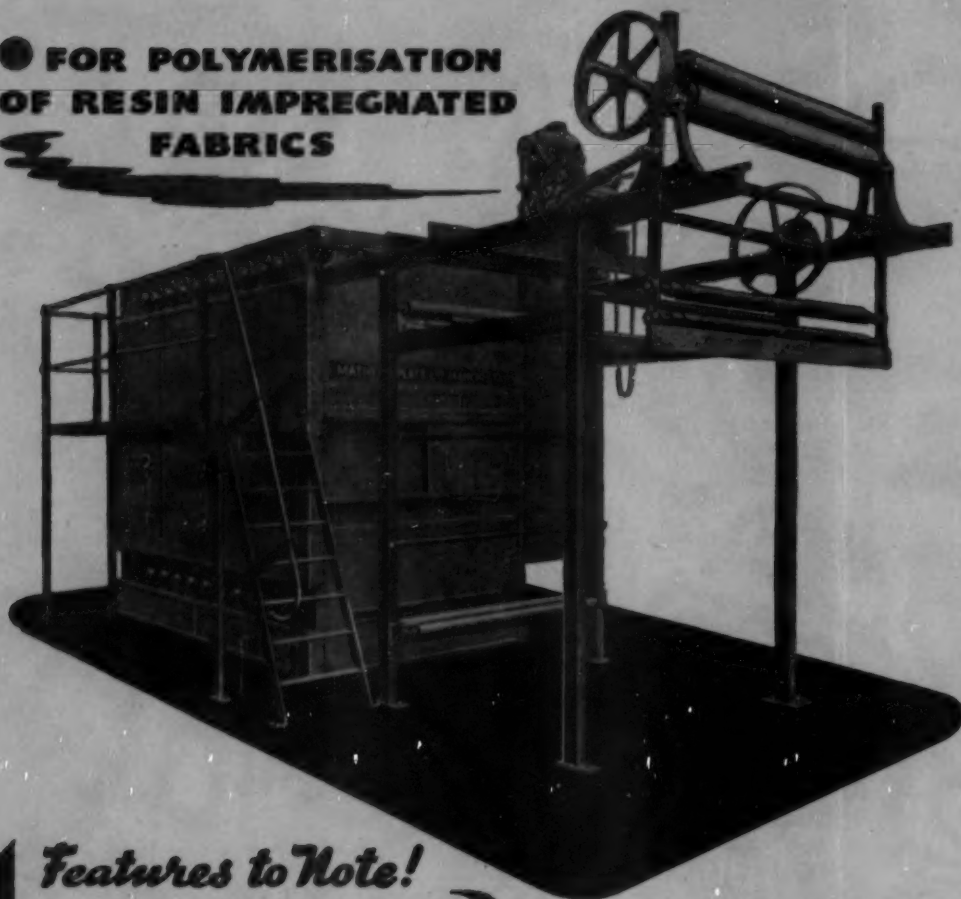
Volume 70      Number 6

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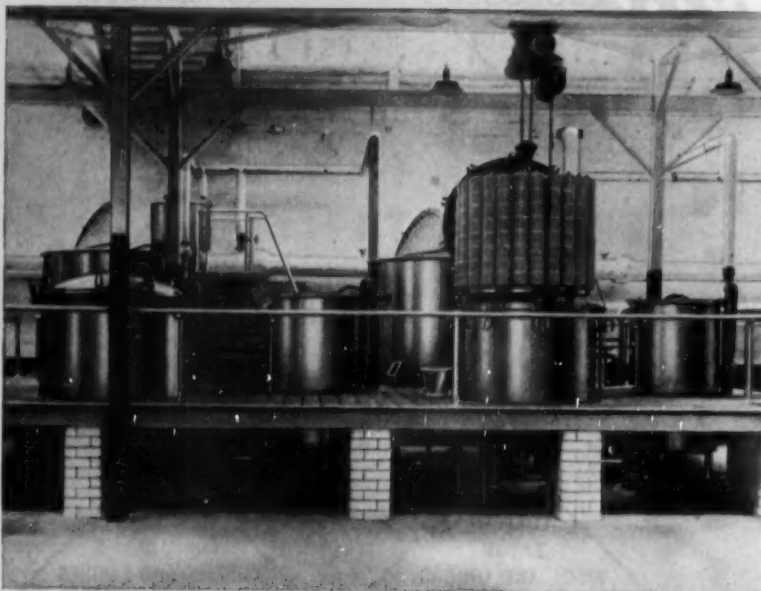


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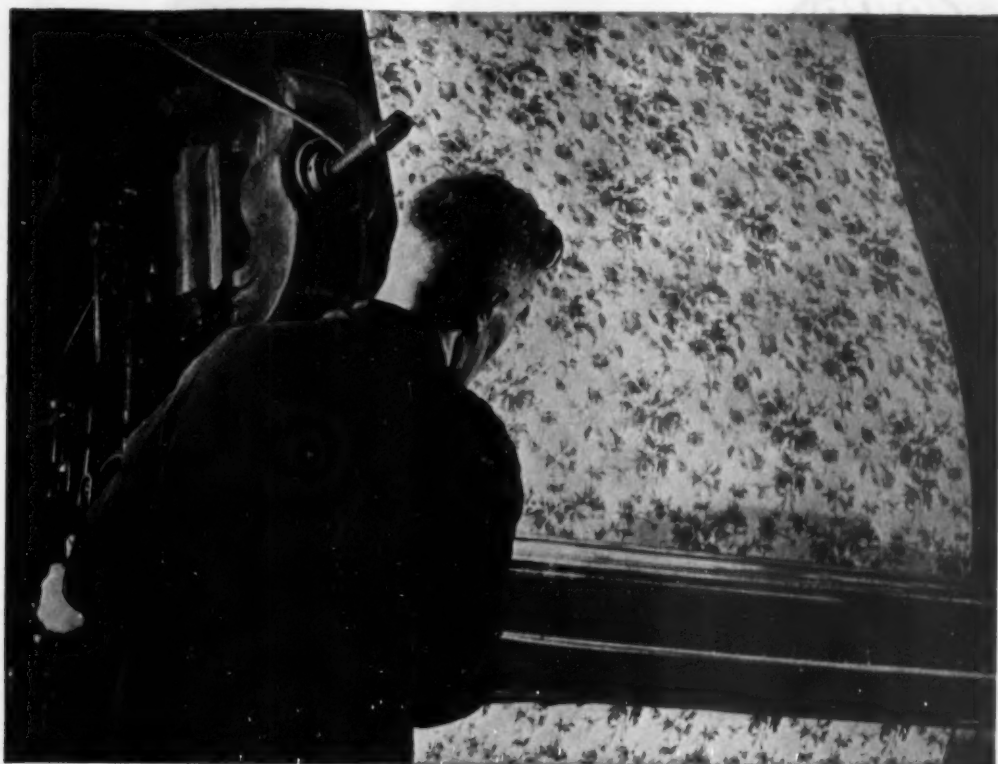
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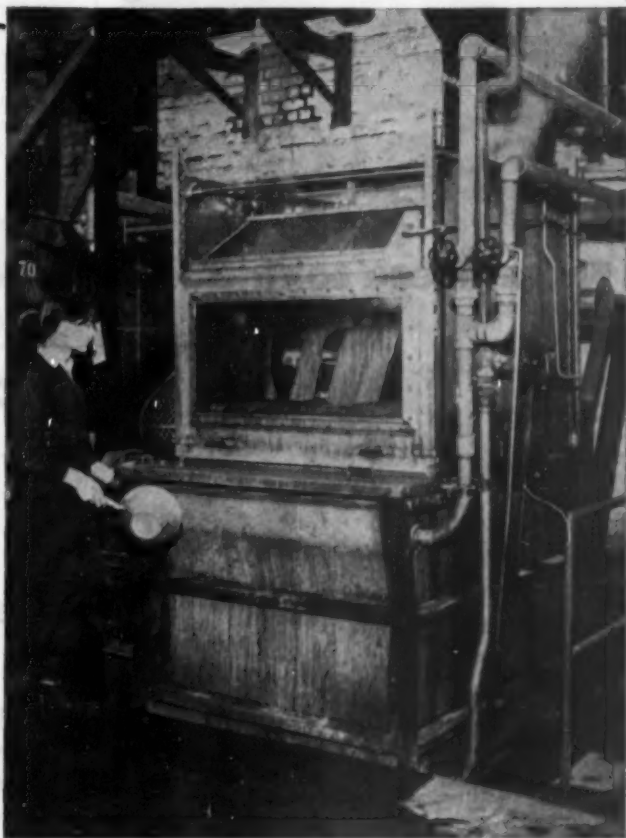
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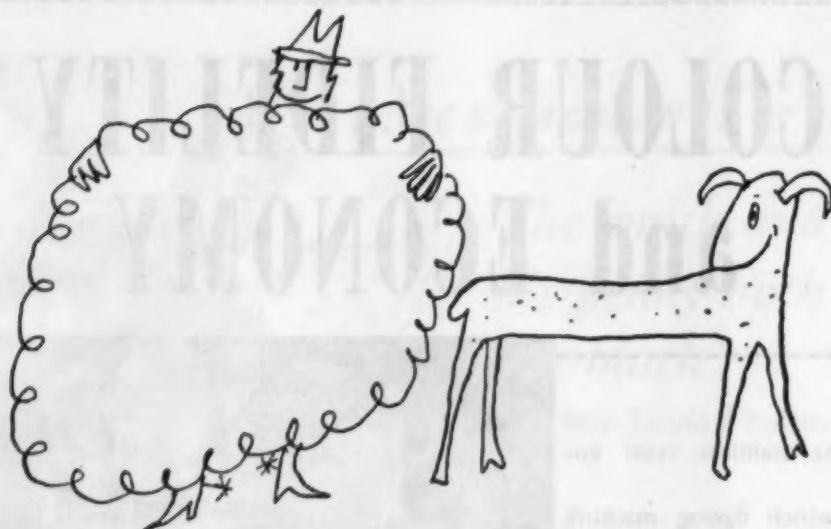
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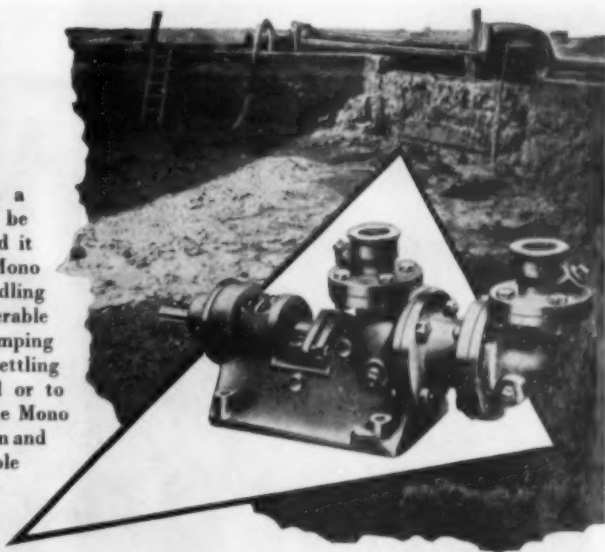
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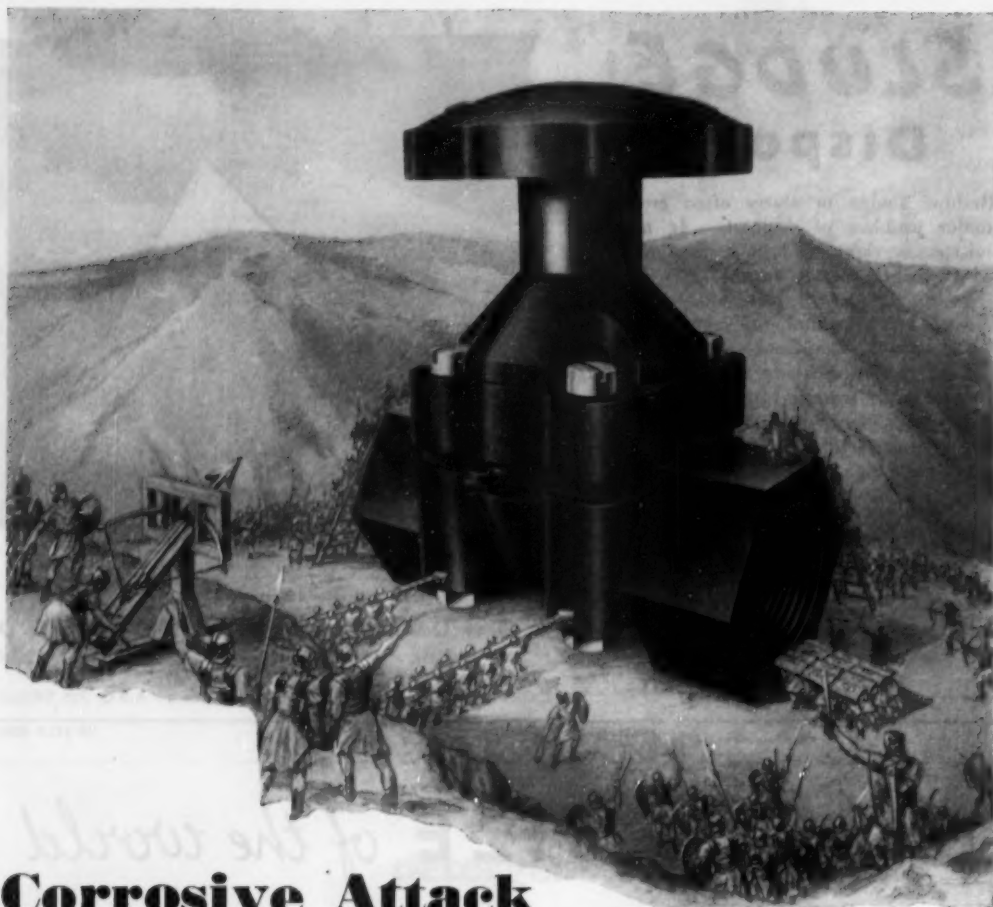
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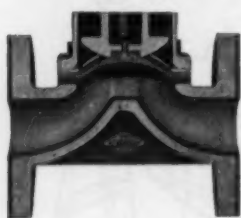
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Readers requiring *general information* regarding the Official Notices, List of Officers of the Society, etc. should consult pages 1-6 of the January 1954 and pages 229-232 of the July 1953 issues of the *Journal*, or write to *The Secretary*, The Society of Dyers and Colourists, Dean House, 19 Piccadilly, Bradford 1, Yorkshire (Telephone Bradford 25138-9). *Editorial Communications* should be addressed to *The Editor*, at the same address.

## Forthcoming Papers

The following papers have been accepted by the Publications Committee, and will appear in future issues of the *Journal*—

### LECTURES

Some Aspects of the Chemistry and Textile Uses of Sequestering Agents *J. V. Summersgill*

Application of Radioactive Tracer Techniques to Textile Research *H. J. White*

### COMMUNICATIONS

The Polarography of Azo Dyes *J. de O. Cabral and H. A. Turner*

The Combination of Wool with Acids *L. Peters and J. B. Speakman*

A Laboratory Apparatus for Detergency Studies *W. A. Straw*

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# THE JOURNAL OF THE Society of Dyers and Colourists

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Volume 70 Number 6

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## Proceedings of the Society Variables in Padding Processes

R. W. SPEKE

*Meetings of the Northern Ireland Section held at the Royal Avenue Hotel, Belfast, on 8th November 1953, Mr. J. Montgomery in the chair; and of the Midlands Section held at the Black Boy Hotel, Nottingham, on 2nd December 1953, Mr. A. P. Kershaw in the chair*

An outline is given of the salient features governing the design of pad mangles for the application of dyes and finishing agents to woven textiles. The important manipulative variables governing the padding process—namely the effects of cloth preparation, running speed, temperature of the pad liquor, and padding oils—are described. The basic principles involved when wet fabrics are repadded are briefly surveyed.

Dyeing techniques involving mechanical impregnation of a fabric with a dye suspension or solution, as far as possible at a stage of no affinity, followed by a fixation treatment *in situ*, are becoming increasingly common. This is largely a result of the modern trend towards continuous processes, which are ideally based on the impregnation → fixation sequence. In addition, the enhanced quality of dyeings obtainable by prepadding in conventional dyeing systems, particularly with heavy or tightly woven fabrics, is leading to its adoption in many new fields. Furthermore, the application of a wide range of textile-finishing agents which have little or no affinity for the substrate is most conveniently effected by mechanical impregnation.

The process of mechanical impregnation is known as *padding*, and usually involves passage of the fabric in open width through a solution or suspension known as the *pad liquor*, followed by a squeeze or extraction to remove excess liquor and to force the pad liquor into the interstices of the fabric and composite yarn structures. Where localised effects must be preserved, e.g. in resist printing or back-filling, the passage through the liquor is dispensed with, the fabric entering the nip directly, and liquor being provided by a furnishing roller running in the thickened pad liquor. It is not proposed to discuss *padding in the nip*, as this process is called, in this paper.

The conventional method of extraction involves the use of positively loaded driven rollers. These rollers, coupled with the necessary impregnation trough, cloth guides, and batching arrangements, form the unit termed variously a *pad*, *pad mangle*, *mangle*, or *padder*.

Normally, dry conditioned fabrics are impregnated, but in certain processes of dyeing and finishing it is desirable to repad a fabric which is already wet.

The successful operation of a pad mangle depends primarily upon the design of the unit itself, coupled with an understanding of the effect of manipulative variables. It is therefore proposed to review the salient features in design and manipulation primarily in connection with the padding of dry fabrics, and to conclude with a short review of a recent study of the mechanism of the repadding of wet fabrics.

### (1) Construction of the Padder

#### 1.1—TENSIONING, GUIDING, AND OPENING DEVICES

The cloth should be fed to the pad trough either from a preformed batch or from a plait—the latter is used almost invariably in continuous processes. Suitable arrangements of guiding, opening, and tensioning devices are necessary, but the design is not critical. It must be realised, however, that, while the application of tension minimises crease formation, it also makes the fabric more difficult to penetrate with the pad liquor.

#### 1.2—THE PAD TROUGH

The pad trough should be designed to give the smallest possible volume of liquor coupled with the longest possible cloth passage. This can be achieved by blanking off the dead space in the trough. With such troughs a 15-in. liquor passage can be obtained on a 60-in. wide machine with an effective liquor volume of 2–3 gal. The use of small troughs avoids wastage at the end of a run, and, what is probably more important, minimises tailing effects when padding with dyes which have an affinity for the

substrate<sup>1</sup>. Small troughs, however, tend to give rise to difficulties where frothing of the dye liquor is encountered. The longest possible liquor passage is clearly desirable to facilitate wetting of the fabric with the pad liquor.

The liquor should be supplied to the trough by a perforated pipe (the perforations pointing away from the cloth) running the whole width of the trough. All pad liquors should be strained, preferably as they are delivered to the trough. Wire mesh sieves, containing 150-300 holes per linear inch, are much more efficient than the rather ill defined filter cloths, some of which are far too porous for effective straining. With troughs of small volume a constant-level device is essential, as the rate of delivery, and hence the effective immersion length, are far more critical than with large troughs. Recirculation of the liquor within the trough is a sound principle to avoid stagnant areas beyond the width of the cloth. An extra installation of this type is not necessary, however, provided the width of the pad trough does not greatly exceed the width of the cloth being processed. Recirculation of the liquor back from the trough to the stock tank is a bad principle, as it means in effect that one is padding through the whole volume of liquor contained in the stock tank.

Naturally, all troughs should be fitted with a free-running immersion roller of the largest possible diameter, and the whole system devised to give ease of access for cleaning, rethreading, etc.

If jacketed troughs are used, the jacket should terminate well below the liquor level to avoid drying out of the pad liquor on the sides of the trough, a defect which can lead to dye specking.

### 1.3— THE NIP

From the pad trough the cloth should travel to the nip itself, meeting the contour of the bowls tangentially. The simplest way of ensuring the correct route is the use of a "horizontal bar". With some fabrics which crease readily (e.g. light-weight staple rayons), a curved expander may be necessary in this position—the curvature should not be too great, however, or uneven wetting may result.

The nip rollers themselves should be built up on solid cast-iron centres covered with a layer of rubber, stainless steel, brass, etc., and the whole mounted on a rigid framework. As the pressure, in conventional mangles, is applied at the ends of the rollers, a bending moment is introduced which may cause the rollers to squeeze more efficiently at the edges than in the centre. This difficulty is accentuated as the width of the rollers and the applied load increase. One manufacturer<sup>2</sup> quotes the roller diameters shown in Table I for machines of different width. Compensation for bending (where encountered) can be provided by grinding a slight camber or crown on the bowls, the exact amount of which required can be determined only by trial. For example, a 50-60-in. padder with roller diameters of 18-20 in. operating on 10 tons load may require a camber of  $\frac{1}{8}$ — $\frac{1}{4}$  in.<sup>3</sup>

TABLE I

Cloth width, in. ...	48	60	72
Roller diameter, in. ...	13.5	15.5	17.5

The covering of the rollers is one of the most important factors in ensuring efficient operation. Even so, many and varied are the types of coverings encountered—stainless steel, vulcanite, brass, soft rubber, etc.—and many combinations of these are found in the individual rollers of mangles.

The factors influencing the choice of covering may be summarised as follows—

- (a) The driven roller normally requires the most durable covering, e.g. stainless steel, brass, or vulcanite.
- (b) Too great a contrast in the hardness of the composite bowls, e.g. stainless steel-soft rubber, is undesirable, as it may lead to two-sided effects with certain fabrics.
- (c) A nip formed by two comparatively soft-covered rollers naturally deforms to a greater extent under any given load, thus reducing the absolute pressure. On the other hand, the greater the nip width the longer is the period during which pressure is applied to the fabric. The greater resiliency of this set-up is an advantage when dealing with the softer, more open weaves, and where surface irregularities have to be dealt with, e.g. some qualities of linens, staple rayons, etc. Conversely, the use of a harder nip, e.g. stainless steel-vulcanite, by developing a greater absolute pressure is more useful when tightly woven structures are to be penetrated, e.g. mercerised cotton poplins, reps, tackle twills, etc., or for water mangling.
- (d) The softer the rubber covering employed the more liable is the bowl to wear during use, necessitating frequent regrounding. Bowls covered with soft rubber also tend to become progressively harder during their useful life. Any rubber covering chosen must therefore effect a compromise between softness and durability.
- (e) The maximum use of metal (especially stainless-steel) bowls facilitates cleaning down the mangle between runs.

Where rubber-covered rollers are employed, it is imperative for maximum efficiency of operation to ensure that the rubber covering is at least  $\frac{1}{8}$  in. thick. With the working load applied, the bowls should deform to give a nip width of at least  $\frac{1}{2}$  in. Nip widths as high as 1.7 in. have been reported in the literature for commercial mangles<sup>4</sup>. The width of the nip and its uniformity can be tested very simply by taking carbon paper impressions, and this provides an extremely quick and useful method of checking mangle performance without carrying out actual dyeing trials.

The hardness of rubber may be measured in many ways, the use of the Shore Durometer, for example, providing a simple method of measurement (Table II). A rubber of 75-85 Shore Durometer reading gives a good compromise between resilience and durability.

TABLE II

## Hardness of Rubber

Shore Durometer Reading	Hardness of Bowl Covering
60-70	Soft
75-85	Moderate
90-100	Hard

It is frequently difficult to decide, when planning the installation of a pad mangle for any specific purpose, whether a two-bowl (giving 1 dip and 1 nip) or a three-bowl (giving 2 dips and 2 nips) is necessary<sup>5</sup>. Normally, if a fully absorbent cloth is to be padded at comparatively low running speeds (less than about 40-50 yd./min.), an efficient two-bowl mangle will prove to be adequate. If, however, the converse applies (i.e. the cloth is not fully absorbent, either owing to inadequate preparation or from the nature of its construction, and/or high running speeds are to be employed), a three-bowl mangle is to be preferred. The three-bowl mangle involves the use of a larger volume of pad liquor, which is a disadvantage when "dyeing in the pad" can take place. The expressed liquor from the second nip falling on fabric as it leaves the first nip has also been known to give rise to uneven dye distribution with certain types of fabric.

Various methods are available for applying the load to the rollers, including dead-set, lever-loading, spring-set, pneumatic systems, and hydraulic devices. The modern trend, however, is towards the use of pneumatic pressure cylinders which are connected to the bowls through a lever system. Independent controls for regulating the pressure to both ends of the rollers are a useful refinement which can be applied with this system. Normally loads of 6-10 tons are applied, giving an absolute pressure at the nip of the order of 200-300 lb. per sq. in.<sup>4</sup>.

## 1.4—THE EXIT ARRANGEMENTS

Finally the cloth should leave the nip tangentially, although following the periphery of the bowls is not as serious a defect on the exit side as on the entry side. If the cloth is then to be batched up, as in discontinuous processes, the batching device should be fitted with an efficient friction clutch in order to avoid putting excessive tension on the cloth as the batch diameter increases. The drive to the batching device should be smooth in its operation to avoid snatch.

## (2) Manipulative Variables in the Padding of Dry Fabrics

The salient features of apparatus design having been discussed, it is now necessary to examine the other variables which influence the quality of the result with any given pad mangle of a conventional type.

## 2.1—CLOTH PREPARATION

When it is realised that the total time of treatment (from entering the liquor to leaving the nip) is only of the order of 2-4 sec. when fabrics are padded at as low a speed as 30 yd./min., it will be appreciated that very rapid wetting of the fabric with the pad liquor is an essential feature of all padding processes. One of the best methods of

ensuring rapid wetting is to give the cloth an adequate preparatory treatment to make it fully absorbent. The great differences in padding behaviour encountered with fabrics which have received different preparatory treatments have been described by Wolfram and Nuessle<sup>4</sup> (Table III relates to a cotton twill).

TABLE III

Preparation	Pick-up (%) <sup>a</sup> after the following dips and nips—		
	1	2	3
Partly desized only ...	23	43	53
Desized, boiled off, and bleached ...	58	64	66

<sup>a</sup> Measured with water only at 25°C.

It is also essential that, whatever preparatory treatment is given, it should be uniform, since, clearly, differences in absorbency will result in uneven pick-up of the pad liquor.

Drying a "rewetting agent" into the fabric prior to padding is one of the most effective ways of increasing the absorbency of the fabric, as shown in Table IV. The data quoted in this table were obtained with a mercerised cotton gaberdine.

TABLE IV

Preparatory Treatment	Pick-up (%) at 50°C. with the following concn. of Calsolene Oil HS (c.c. per litre) —				
	0	2.5	5.0	10	20
Desized only ...	14	27	34	46	60
Desized, dried, padded with Calsolene Oil HS, and dried *	50	52	54	58	59

\* 10 c.c. Calsolene Oil HS per litre at 90°C., 2 dips + 2 nips, pick-up approx. 50%.

Unfortunately, unless the fabric is dried before impregnation with the Calsolene Oil HS, it is difficult to ensure an even distribution of the rewetting agent in the fabric (see §3). In continuous processing, particularly with cotton goods, some form of continuous cloth preparation prior to dyeing ultimately becomes a necessity on both economical and technical grounds. For this reason, many of the preparatory treatments employed are not really adequate for subsequent high-speed padding processes, and the development of a suitable continuous system of cloth preparation before dyeing is long overdue.

## 2.2—RUNNING SPEED

An increase in the running speed effects the following—

- The time of immersion in the pad liquor decreases: hence there is less time for complete and uniform wetting of the fibre to take place, but when dyes which have an affinity for the fibre are being padded, there is less chance of "dyeing in the pad".
- The "squeezing time" decreases: hence complete penetration of the fabric structure is less likely to be obtained; the decreased squeezing effect also results in a higher pick-up being obtained at the higher running speeds with all absorbent fabrics.

In batch-wise processing, speeds of up to 50 yd. per min. are most commonly encountered; in continuous processing (e.g. pad-steam process), however, speeds of up to 80–100 yd./min. are more normal.

### 2.3—TEMPERATURE OF PAD LIQUOR

As the temperature of the pad liquor is raised, it becomes progressively easier to obtain more rapid and more uniform wetting of the fabric. The effect of temperature on pick-up with a cotton drill (both loom-state and fully prepared) with a variety of padding assistants is shown in Table V.

TABLE V

Pad Liquor (per litre)	Pick-up (%) at—			
	20°C.	50°C.	70°C.	90°C.
LOOM-STATE				
Water ... ..	17	25	26	24.5
10 c.c. Calsolene Oil HS ...	43	47	49	50
5 g. Permal PP ... ..	51	52	50	49
10 c.c. Lissapol N ... ..	44	44	41	39
DESIZED AND SCAURED				
Water ... ..	54	71	75	72
10 c.c. Calsolene Oil HS ...	75	77	72	66
5 g. Permal PP ... ..	79	77	72	68
10 c.c. Lissapol N ... ..	77	72	71	64

Higher padding temperatures are also to be preferred where soluble dyes are being employed, e.g. with direct cotton dyes, Soledons, Brenthols, reduced sulphur dyes (or the water-soluble Thionol M brand), as they assist the preparation and maintenance of true solutions at high concentrations.

Where vat dye dispersions are involved, the higher the temperature of the pad liquor the more liable is aggregation or flocculation of the dispersion to take place (see § 2.4). With this type of dye, therefore, a compromise must be made between the beneficial and adverse effects of temperature, padding being normally effected at 50–60°C.

A minor point which requires attention on prolonged runs when high padding temperatures are being employed concerns the avoidance of evaporation of the pad liquor. When a large bulk of liquor is made up and stored for long periods in open tanks, an appreciable amount of evaporation can take place, leading to a build-up in colour.

### 2.4—PADDING OILS

Normally an addition is made to the pad liquor of a padding assistant. These so called "padding oils" should exhibit the following characteristics—

#### (a) High-speed Wetting Efficiency

This is the primary function of any such assistant. High-speed wetting of the fabric by the pad liquor enables the condition of equilibrium wetting to be approached in the limited time available, and hence improves the uniformity of the impregnation. The preferred method of testing the wetting efficiency of agents of this type is to measure the pick-up obtained by padding a loom-state fabric through wetting agent solutions of increasing concentration, the maximum test concentration employed being well in excess of that normally employed (e.g. 30 c.c. Calsolene Oil HS per

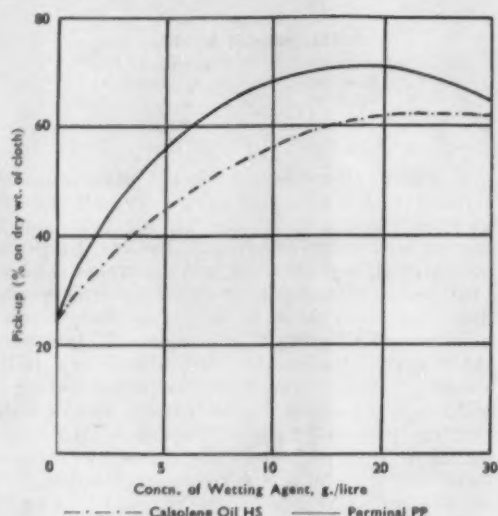


FIG. 1—Wetting Efficiency by Padding at 50°C. (loom-state cotton drill)

litre). This enables relative comparisons in concentration efficiency to be made under given padding conditions. Fig. 1 illustrates the results obtained by this method in comparing Calsolene Oil HS with Permal PP. On the basis of this test, Permal PP is stronger, approx. 45 : 100 against Calsolene Oil HS.

As pointed out by Wolfram and Nuesale<sup>6</sup>, who first advocated this method of test, the use of yarn-sinking tests (such as the Draves-Clarkson and the Herbig tests) is valueless for the assessment of wetting efficiency under padding conditions, because both the fundamentals of the wetting action and the concentration range differ widely. For example, a comparison of Permal PP with Calsolene Oil HS made by the Herbig test showed the Permal PP to be stronger than the Calsolene Oil HS in the ratio of 62.5 : 100 compared with 45 : 100 by the loom-state fabric test.

In all cases the use of excessive concentrations of wetting agent should be avoided. On fully absorbent fabrics increase in wetting agent concentration decreases slightly the pick-up of pad liquor, and decreases appreciably the ultimate apparent colour yield obtained. Tests have shown<sup>7</sup> that the decreased colour yield obtained on absorbent fabrics with excessive quantities of wetting agent is entirely a function of the increased penetration of dye into the fabric (and composite yarn) structures.

Experience has shown that, in translating laboratory padding recipes to bulk scale, the use of a padding assistant enables better reproducibility to be obtained. This is due to the fact that, even after allowing for expression, bulk-scale mangles may produce a better penetration than laboratory mangles. The presence of padding assistants helps to minimise this difference.

#### (b) Freedom from Excessive Frothing

The rapid passage of cloth through the pad liquor coupled with the expression of liquor and air

from the fabric at the nip gives ideal conditions for foam formation. The tendency for certain padding assistants to promote foam formation is well known. This property can be assessed in the laboratory by agitating a known volume of wetting agent solution in a measuring cylinder under standard conditions and measuring both the volume of foam produced and its stability. For example, a comparison of Permal PP and Calsolene Oil HS shows the latter to be slightly better than the former in this respect (Table VI).

TABLE VI

Assistants	Volume of Foam * (c.c.) after (sec.)—						
	0	15	30	60	120	240	300
Calsolene Oil HS ...	45	25	15	0	—	—	—
Permal PP ...	55	25	10	10	10	5	0

\* Produced by agitation of 25 c.c. of soln. at 50°C. in a 250-c.c. measuring cylinder with 40 strokes of a rubber plunger.

### (c) No Adverse Effect on Dyes

Padding assistants must always be chosen with an eye to the nature of the dyes and the subsequent fixation treatments employed. For example, cationic wetting agents will coprecipitate with anionic dyes, and ethylene oxide condensates will exert a restraining effect during the subsequent fixation of vat dyes.

One of the most common difficulties of this type is encountered with vat dye dispersions in the presence of anionic padding oils. Aqueous suspensions of the highly dispersed forms of pigment vat dyes marketed especially for padding processes may be cracked or flocculated under the combined effects of—

- (i) Presence of padding assistants, the nature and the concentration of the assistant being important.
- (ii) Storage, for periods of a few hours only
- (iii) High temperatures.

Fox<sup>a</sup> has studied the dispersion stability of a range of Caledon dyes for periods of 1 hr. at 50° and 80°C. in the presence of 10 c.c. Calsolene Oil HS per litre. The dyes examined were classified into three groups (Table VII). With the exception of the dyes marked with an asterisk (\*), the dispersion stability was assessed with FD Caledon Powder Fine dyes. In general, the FD brands were found to be slightly more sensitive than the corresponding Paste Fine brands. It must be appreciated that the test conditions and method of assessment employed were designed to be severe.

TABLE VII

### A—Caledon Dyes appreciably aggregated at 50° and 80°C.

Gold Orange 3G	Brilliant Purple 4R
Red 4B	Olive Green B
Brown R	Blue GCP
Brown G	Dark Blue 2R
Olive R	Violet XBN
Dark Brown 3R	Green 2B
Brown 3G	Black 2BM
*(Alizaranthrene)	Direct Black R
Navy Blue RT	Grey M

### B—Caledon Dyes appreciably aggregated at 80° but not at 50°C.

Brilliant Orange 6R	Brilliant Violet R
Red BN	Brilliant Violet 3R
Brilliant Red 3B	Blue XRN
Dark Brown 6R	Brilliant Blue 3G
Dark Brown 2G	Dark Blue G
Khaki 2G	*(Alizaranthrene)
Khaki R	Navy Blue R
	Direct Black AC

### C—Caledon Dyes showing No Significant Aggregation at 50° or 80°C.

Yellow GN	Blue XRC
Golden Yellow GK	Blue GXD
Red 5GD	Jade Green 3B
Pink RL	Jade Green XBN
Red Violet 2RN	Jade Green 2G

A product has been developed, named *Permal PP*, which gives far less aggregation than Calsolene Oil HS under the combined effects of high temperature and storage. With the exception of Caledon Red 4B, none of the dyes listed in Table VII, with up to 5 g. Permal PP per litre, showed any significant tendency to aggregate after storage for 1 hr. at 80°C.

It must be emphasised that, even when Permal PP is used, aggregation may be encountered if excessive concentrations are employed or if storage times and temperatures in excess of 1 hr. at 80°C. are employed. In fact, the only safe way to carry out long runs when pigment-padding at high temperatures is to prepare the bulk of the liquor at room temperature and to preheat it as it is delivered to the pad trough.

Permal PP is in no way designed as a general replacement for Calsolene Oil HS, but should be used in special cases where difficulty has been encountered or where high temperatures or long storage periods are involved.

### (3) The Repadding of Wet Fabrics

Occasionally the repadding of wet fabrics is necessary to avoid a costly intermediate drying stage, e.g. in the continuous application of Soledon dyes, or in the process pigment-pad → (wet) → chemical-pad → steam. Before repadding it is obviously necessary to give the fabric a uniform squeeze, and empirically it is clearly beneficial to obtain the highest possible liquor extraction at this stage.

Considering, then, the second padding process, let us imagine that the cloth enters the padder carrying  $P\%$  of liquor and emerges from the second nip carrying  $C\%$  of liquor (see Fig. 2). The quantity  $(C - P)\%$  is defined as the *pick-up difference*. During passage of the cloth through the second nip a certain amount of the first pad liquor will be displaced, and let us imagine that on emergence the cloth carries a quantity  $p\%$  of the first pad liquor. The amount of the first pad liquor displaced will then be  $(P - p)\%$ . This factor is called the *interchange value*. The true pick-up of the second pad liquor will be  $(C - p)\%$ .

The values of  $C$  and  $P$  can be measured simply by direct weighings. The value of  $(C - p)$  can be measured by first padding through water and then repadding through a solution of a non-substantive acid dye such as Azo Geranine 2G, which can then be extracted quantitatively and measured.

In practice, at the second padding stage it is obviously desirable to—

- Obtain the highest possible true pick-up of the second pad liquor
- Keep the interchange value at a minimum to reduce contamination or dilution of the second pad liquor
- Compensate for dilution effects introduced by the displacement of the first pad liquor at the second nip.

A good guide to the realisation of these requirements can be obtained by studying results obtained in the laboratory using the Azo Geranine test described above, as shown in Table VIII.

the second padding in these circumstances. Usually it is more important to keep the interchange value at a minimum to avoid contamination of the second pad liquor, and hence a two-bowl mangle is employed.

(c) Dilution of the second pad liquor by the interchange liquor can be allowed for by feeding a stronger solution than was originally present in the trough. It can be shown that the relative strengths of the two solutions is given by the formula—

$$\frac{\text{Feeding concentration}}{\text{Starting concentration}} = \frac{\text{True pick-up}}{\text{Pick-up difference}}$$

The magnitude of this ratio is shown in the final column of Table VIII. The problem of dilution is

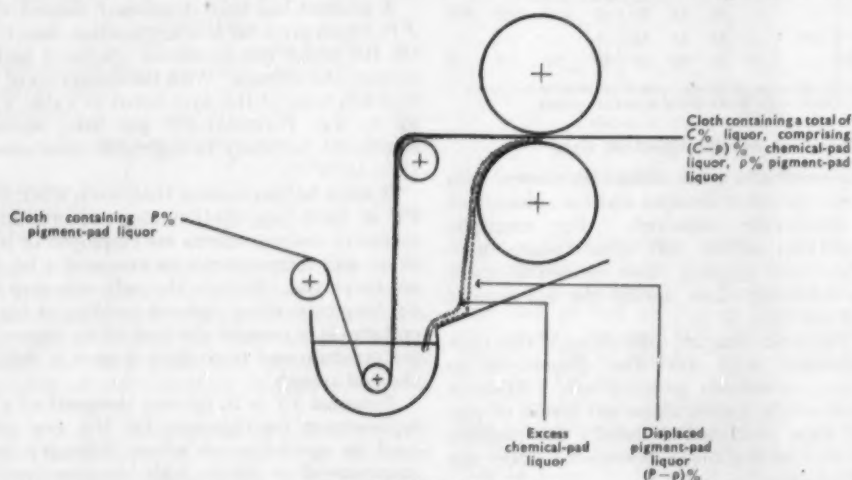


FIG. 2—Repadding of Wet Fabrics

TABLE VIII

P%	C%	(C-P)%	(C-p)%	(P-p)%	$\frac{C-p}{C-P}$
A— WITH A TWO-BOWL PADDER FOR THE SECOND MANGLE					
65	65	0	25.4	25.4	—
65	75	10	30.5	20.5	3.1
65	85	20	35.5	15.5	1.8
65	95	30	40.5	10.5	1.3
B— WITH A THREE-BOWL PADDER FOR THE SECOND MANGLE					
65	65	0	38.8	38.8	—
65	75	10	44.2	34.2	4.4
65	85	20	52.6	32.6	2.6
65	95	30	60.4	30.4	2.0

A study of these data shows that the solution to the three practical problems described above is as follows—

- True pick-up of the second pad liquor ( $C - p$ ) increases as the pick-up difference ( $C - P$ ) increases. With any given pick-up difference, a much higher pick-up is obtained with a three-bowl padder than with a two-bowl mangle.
- The interchange value ( $P - p$ ) decreases as the pick-up difference increases. With any given pick-up difference a much higher interchange value is obtained with a three-bowl padder than with a two-bowl padder. Two mutually opposing factors therefore govern the choice of mangle to be used for

obviously minimised by the use of a two-bowl mangle operating with the maximum pick-up difference. A more detailed mathematical treatment of such effects has been reported by Marshall<sup>1</sup>.

#### (4) Conclusion

In conclusion, it may be said that the pad mangle is now a necessity in all modern dyeworks. The efficient operation of this unit demands a basically sound construction utilised with an intelligent understanding of the influence of the possible manipulative variables.

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(MS. received 9th February 1954)

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## Colour Fastness and Textile Finishing Requirements of the Ultimate Consumer

J. S. INGHAM

*Meetings of the Scottish Section held at the St. Enoch Hotel, Glasgow, on 17th November 1953, Mr. R. D. Alexander in the chair; of the Manchester Junior Branch held at the College of Technology, Manchester, on 10th November 1953, Mr. J. Boulton in the chair; and of the Huddersfield Section held at Field's Cafe, Huddersfield, on 16th March 1954, Mr. E. Rolfe in the chair*

A large retail organisation quickly obtains the public's reaction towards visible factors of style and colour. During the past few years, we have attempted in our laboratories to obtain some information on the invisible factors of durability and wearability. From a survey and our laboratory findings, it is clear that certain standards must be achieved to meet the demands of the public. This paper deals with some of these findings and observations in the field of dyeing, printing, and finishing. Further, we have tried to pass on the information to dyers, finishers, and dyemakers, and pointed out the gaps in production, which it would be very desirable to fill to meet the public's demand.

In order to maintain a nation-wide retail distribution business in a flourishing condition, several basic merchandising principles must be applied—

- (1) The price must be right
- (2) The design, style, and colour must be what the customer wants
- (3) The quality must be the best possible at that price.

We consider that, in order to ensure customers' satisfaction as far as such factors as wearing properties and durability are concerned, it is essential for us to maintain close relationships with suppliers at all stages from the raw material to the finished article. In this way we, who are closest to the consuming public and can assess its requirements, are enabled to transmit them to spinners, weavers, knitters, dyers, finishers, and printers. With such contacts we are able also to keep abreast of newer developments in the textile industry, and thus can translate these new ideas into merchandise of better and better quality for the mass public.

This paper is confined to the control of quality, and the implementation of research and development within the dyeing, printing, and finishing sections of the textile industry in improving the quality of merchandise that we sell.

It was necessary for us to attempt to establish minimum performance standards, and on woven cloths we used the Utility specifications<sup>1</sup> as a yardstick. In our laboratories we attempted to correlate customers' returns with the results of physical tests. The Utility schedule was an admirable system for the most economical utilisation of raw materials and labour in the production of fabrics which would give good service, but, because of the fact that it rigidly laid down cloth construction and certain standards of dyeing and finishing, it tended over the years to stifle development. During its operation, however, our laboratory staff were able to pinpoint weaknesses in cloth construction and establish certain minimum performance standards, which were evolved from an examination of customers' returns.

It must be emphasised that it is sometimes difficult to assess what is a "true" customer's return. In this connection, the admixture of strong

alkalis and oxidising and bleaching agents in some of the detergents sold in this country, and also the claims made by some of the manufacturers of them, are to be strongly deprecated. We also now have to contend with the use of "optical bleaching agents" (fluorescent whitening agents) in these preparations. Some of these bring about the most amazing colour changes, even to vat dyes, and they turn yellow on exposure to sunlight or heat, particularly under alkaline conditions. We have also to take into account the increasing use of rotary washing machines.

The "genuine" customers' returns, although undesirable, are of value to us. It is, of course, imperative to cut them to a minimum. For every customer who returns a garment when there is some cause for complaint, there may be ten who do not but who are, nevertheless, dissatisfied to the extent that they will probably not buy any more.

Among performance standards established by this work are the following—

- (1) The strength of a raised winceyette or flannelette for a 2-in. strip on the Scott machine should not be less than 40 lb.
- (2) The wet strength of a rayon staple or filament cloth should not be less than 16 lb. for a 2-in. strip on the Scott machine.
- (3) On our slippage test<sup>2</sup> minimum performance standards for filament cloth slippage have been established.

### COTTONS

The Utility schedule for coloured woven flannelles specified that the dyes used should pass the No. 2 washing fastness test of the Society of Dyers and Colourists<sup>3</sup>. This meant that the classes of dyes used were diazotised and developed, after-coppered and copper-complex dyes, aftertreated directs, and, in some cases, direct cotton dyes alone. Experience showed that this standard was not sufficiently high to meet public demand.

Later, Ciba Blue was used along with some sulphur dyes, but the majority of dyers used primuline for the reds. This last dye gave a great deal of trouble because of its lack of washing fastness, although it is significant that we had no

returns because of poor light fastness on such garments. At the present time, therefore, we have specified vat dyes and azoic reds. The latter are presenting a certain amount of difficulty because of staining of adjacent whites during processing.

The ground shades on printed raised cloths leave something to be desired, because many dyers are padding, as distinct from dyeing, and the steaming time is insufficient for fixation. This applies particularly to a dye such as Orange WS.

Our principal difficulty on corduroys and velvets is to produce reds and maroons of reasonable fastness to washing which are also fast to dry and wet rubbing. There is an urgent need for a solution to this problem.

We prefer boiler suits to be dyed with vat dyes rather than sulphur dyes.

There is a demand for improving the fastness of vat blues on shirtings to the action of oxidising agents such as chlorine. Again, it cannot be overstressed that fluorescent compounds play havoc with many of the plain vat shirting colours.

#### RAYON STAPLE (SPUN RAYON)

We have had some trouble because of uneven yarn, which has resulted in a "warped stripy" effect, and the use of sizes which are difficult to remove has also contributed to the problem. The effect has certainly not been caused by viscoses of different ageings. This uneven effect was particularly pronounced on dischargeable ground shades for prints. Pad dyeing does help, provided that fastness can be maintained.

We are experimenting with the use of after-treated direct cotton dyes and azoic reds for children's garments, where there is the possibility of the use of white embroidery or white collars. We have found also that it is essential to use azoic reds for children's buster suits. None of the diazotised and developed reds or aftertreated reds are satisfactory.

Our greatest difficulty with staple rayons, however, is the poor reproducibility of the crease-resisting finish: this finish varies considerably from one finisher to another and from delivery to delivery. There are, of course, many factors which contribute to this, such as the yarn irregularity, the twist, the sizing, the amount of resin, the degree of fixation, the degree of condensation of the pre-condensate, and possibly the time of baking. We have had Utility 1009 cloths with as much as 20% and with as little as 4% by weight of resin. Some are very soft, some are hard, and others sew very badly.

This last causes concern, because breakdown is often not apparent until a garment is washed. From the work of Dorking and Chamberlain<sup>4</sup>, it is clear that some of the sewing problems can be overcome by altering machinery techniques, but production of a consistent finish should be regarded by the finishing industry as a challenge. There does not seem to be any method of assessing whether the cloth will sew properly or not, but, as a very rough guide, we consider that 1009 fabric which gives an abrasion figure of less than 6000 on the Martindale tester, and 1005 fabric which gives

a figure of less than 3000, will give us sewing difficulties or breakdown of the seams on the first washing.

At the present moment, we are reviewing several cloths and are attempting to correlate sewability with design, strength, crease recovery, evenness of treatment (assessed by the Best-Gordon technique<sup>5</sup>), and resin content, but this is very difficult.

#### FILAMENT RAYON

We consider that the decline in popularity of the Utility types of viscose rayons 1050 and 1051 was due to their high shrinkage and to their low strength. The introduction of shrink-resisting treatments has helped filament cloths, but the tendency today seems to be to use fine-filament cloths, which do not shrink to so great an extent as the 1050 types. Again, we require a minimum wet strength of more than 16 lb. for a 2-in. strip.

Care must be taken with the delustring of acetate rayon cloths, but fortunately at present fashion demands a brighter finish than hitherto, with the result that we have not the difficulty of loss of strength caused by delustring. It would appear that, no matter how the delustring is brought about, there is always tendering of these cloths.

One factor which is difficult to explain is why cloths having acetate rayon warps and viscose rayon wefts, say 75-denier wefts, become weak in the weft after printing.

#### WOOLLENS AND WORSTEDS

As a very rough guide, a woollen afghalaine fabric and a velour should withstand 7,000 and 10,000 cycles respectively on the Martindale abrasion tester before two threads break. We consider that a strength of 20 lb. per 2-in. strip is reasonable for afghalaines and 30 lb. for velours. No correlation could be found between the wearer trials and laboratory tests.

Shrinkages under the Hoffman press during garment making have given a lot of trouble. A test was devised in our laboratories as follows—

A test cloth is inserted, the top of the press closed, and steam applied at 60-70 lb./sq.in. for 5 sec. The top is then opened, and the vacuum applied for 5 sec. If the shrinkage is greater than 3% under these conditions, we consider the fabric to be unsatisfactory.

On serges for gym tunics we have eliminated the use of the type of chrome-complex dyes requiring large amounts of strong sulphuric acid, because of the adverse effect of the acid on wearing properties. It was interesting to note that, of our three standard colours, we received returns on the greens, which were dyed with these chrome-complex dyes, and not on the navy blues and nigger browns, which were afterchromed.

Some trouble has been experienced with the poor fastness to rubbing of cotton-warp flannels. Experience has shown that this can be improved if there is a minimum severity of stripping before dyeing, preferably with a complete avoidance of chrome stripping. Secondly, the pieces must be thoroughly washed off at the end of the filling-in process.

As far as dyed worsteds are concerned, particularly for export, where the sponging process is used on the pieces, we have found that the cloth should withstand the American test for fastness to perspiration<sup>6</sup>. The results of this test correlate fairly well with marking off during the sponging process.

#### KNITTED OUTERWEAR

Our principal concern with knitted outerwear has been to obtain worsted yarns and garments of adequate fastness to washing.

Again, an analysis of customers' returns showed that the yarn should withstand the S.D.C. No. 2 Test (Wool)<sup>3</sup> for safety. We do not strictly adhere to the test, because we may accept colour loss in the test provided that there is no staining of the white backing cloth: staining is far more important than colour loss. We had many difficulties in obtaining this standard, but thanks to the co-operation of manufacturers, our dyers, and dyemakers, we have relatively few troubles in obtaining level dyeings with some of the faster type of acid dye and the chrome dyes.

Occasionally, the chrome dyes and some of the neutral-dyeing acid dyes do appear to make the yarn brittle, and consequently there are winding difficulties and apparent lowering in wool quality. It must be stressed that yarn is often sent under-conditioned, not being allowed to condition before waxing and knitting, and consequently the regain of moisture is inhibited, with the result that garments feel hard and brittle. Also, sometimes, when one speaks of "neutral dyeing", the wool may actually be in an alkaline condition at a relatively high temperature during the early stages of dyeing.

#### OTHER TYPES OF LADIES' CARDIGANS AND PULLOVERS

We are shrink-resisting and garment-dyeing single-bed-knitted fabrics. This is very successful, provided that care is taken to ensure that a single dyeing is made up of bodies knitted from yarn of the same blend, quality, and age. Some difficulty is experienced in matching the ribbing and stolling with the body, particularly in pale blues and pinks.

For these garment-dyed fabrics, we insist on fastness to the S.D.C. No. 2 Test (Wool)<sup>3</sup>.

#### JERSEY FABRIC

Most of our jersey fabric now has a decatized finish, because we find that it drapes better and has a better appearance in the stores. We are experimenting to produce a washable jersey fabric, particularly for ladies' and children's separates, but one trouble seems to be to overcome the relaxation shrinkage. Shrink-resisting and fast dyeing of these fabrics also constitute a problem.

Customers' returns on children's matinée coats and pram sets and on ladies' cardigans have been rather serious in cases where we have used fluorescent dyes. One reason for this has been that some dyers have been skimping their bleach and overloading with these agents, but the principal trouble has been caused by yellowing when the garments have been dried (after washing) in direct sunlight or by radiant heat, and we are therefore faced with the possibility of having to eliminate this line.

#### INTERLOCK FABRIC

With interlock and similar types of fabrics, use has been made of spun-coloured rayon mixtures with cotton dyed in the loose before spinning. Satisfactory fastness can be obtained, and hence fairly cheap yarns produced by this method, with, of course, the exception of all reds and some of the dark colours such as navy.

All reds on circular-knit fabric should be azoic-dyed, but the cost of azoic and of vat dyeings tends to put them out of our price range. Diazotised and developed dyeings and aftertreated direct cotton dyeings have helped.

#### MEN'S HALF-HOSE

Our dark solid shades are all chrome-dyed, and it is interesting to note that we have eliminated Coomassie Navy Blue in favour of a chrome navy, and that we have received no customers' returns due to dermatitis. We had some trouble at one period with the matching of the nylon heels and toes, but this seems to have been overcome.

The only mixture half-hose that we are selling are 50 : 50 viscose rayon-wool blends, and there the use of cation-active substances has helped in obtaining the required fastness. We also find that it is necessary to give a shrink-resist treatment to these socks even in the heavy weight.

#### ECONOMIC FACTORS

Some of the customers' requirements have been indicated above. Some can be satisfied, and others are in need of further investigation. Of those which can be fulfilled, it must be pointed out that it is not always economically expedient to do so.

For instance, a strong case has to be presented for increasing the price of a garment at present selling at, say, 19s 11d, because it is certain that sales would decrease considerably if the price were over £1. Thus we feel that our pale direct dyeings for athletic shirts in crease-resisted rayon staple are of adequate fastness, and that it would be of little advantage to use vat dyes, especially as it would mean an increase in price.

Again, the permanent mothproofing of a velour would cost of the order of 6d per yard, which means that the selling price of the garment may be increased by 2s 6d.

These factors should be borne in mind when it is wondered why the processes evolved in the laboratory are not always taken up as quickly as would appear desirable.

MARKS & SPENCERS LTD.

82 BAKER STREET

LONDON W.1

(MS. received 17th February 1954)

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## COMMUNICATION

## The Preparation and Properties of Some Inorganic Alginate Fibres

(Mrs.) M. J. DUDGEON, R. S. THOMAS, and F. N. WOODWARD

Alginate fibres have been produced in which alginic acid is combined partly with calcium and partly with other metals. In the case of all the metals examined (chromium, copper, cadmium, tin, bismuth, barium, zinc, and aluminium) there is a reduction in water imbibition but, with the exception of aluminium, barium, and cadmium, this is accompanied by a reduction in dry and wet extensibilities. Calcium-cadmium alginate appears to be most promising, since, compared with calcium alginate fibre, it shows no loss in dry extensibility, an increased wet tenacity, and only a small loss in dry tenacity, and, moreover, can be produced with no more difficulty than calcium alginate.

Hoagland<sup>1</sup> has shown that the alginates of lithium, sodium, potassium, ammonium, and magnesium are water-soluble, whilst the alginates of other metals and alginic acid itself are insoluble. It should therefore be possible to produce insoluble alginate fibres by using as a base any metal except those mentioned above. It should also be possible to modify the fibre properties by neutralising a part of the alginic acid with one metal and completing the neutralisation with another.

No systematic study on these lines appears to have been made, although the patent literature contains many references to alginate fibres and yarns.

Bonnicksen<sup>2</sup> has described the production of transparent films from calcium alginate, and Rose<sup>3</sup> that of films and fibres from alginic acid. Speakman and his collaborators<sup>4</sup> have pointed out the disadvantages of alginic acid yarn as a textile material, in particular the ease with which it is swollen and dissolved by dilute alkalis. They have shown that this difficulty can be overcome to some degree by aftertreating the alginic acid yarn with hardening solutions of metallic salts such as calcium chloride and potash alum, and particularly with metals of high co-ordinating power such as chromium. Chromium alginate has the disadvantage of being highly coloured, but Speakman<sup>5</sup> later found that uncoloured, alkali-resistant fibres may be obtained by treating alginic acid or calcium alginate fibres with basic beryllium compounds. He also prepared aluminium, barium, iron, and zinc alginate fibres, although their textile properties were not assessed.

During the development of calcium alginate fibre as a camouflage material during World War II, several defects became apparent. These were in part due to variations in the raw material, but could also be attributed to the use of calcium salts to neutralise the alginic acid.

The main defects were—

- (1) A high water imbibition, which made the fibre "soggy" when wet
- (2) A low wet strength compared with the dry strength
- (3) The fibre became very brittle at low moisture contents, i.e. it had a low dry extensibility.

The work described in this paper was undertaken primarily with the object of improving the quality of the fibre for camouflage purposes, particularly in respect of the above properties.

Since considerable experience had been gained in the large-scale production of calcium alginate fibre,

and as calcium was likely to be the cheapest suitable metal, the investigation was designed to cover a range of mixed metal alginates in which calcium was one component. It was thereby hoped to determine the minimum percentage replacement of calcium by other more expensive metals necessary to give the desired improvement. Owing to limitations of time, it was not possible to prepare and evaluate some obvious possibilities, such as manganese and cobalt alginates, nor was it possible fully to assess the properties of even the more promising metal alginate fibres.

The investigation was therefore restricted to a consideration of the influence of certain metals on the water imbibition, dry and wet tenacities, and extensibilities of certain inorganic alginate fibres. The general procedure followed was to produce a calcium alginate fibre and replace part or all of the calcium by aftertreatment of the freshly spun fibre before drying.

## Experimental

The experimental work can be considered under three headings—(a) the preparation of calcium alginate suitable for fibre production, (b) the preparation of the spinning "dope" and the working out of spinning conditions, and (c) aftertreatment of the calcium alginate fibre.

## CALCIUM ALGINATE

It was known that the past history of the calcium alginate and its quality, in particular as judged by its viscosity, sulphur and "weed residue" contents, and also by its content of acid-soluble alginic acid, influenced the properties of the resultant fibre. It was therefore essential to use a consistent supply of good-quality material.

At the time this investigation was started in 1943, large-scale production of calcium alginate was from the littoral brown seaweed *Ascophyllum nodosum*, and a 30-lb. sample of the material was thoroughly mixed to ensure homogeneity, and analysed. The results, as shown in Table I (A), indicate a rather low viscosity and a very low calcium alginate content. A part of the material unaccounted for consisted of calcium carbonate, and part low-polymer alginic acid, which is soluble in *N*. hydrochloric acid.

A trial series of mixed calcium-aluminium fibres (No. 1-5, Table III), including a "blank" calcium alginate fibre, was first produced. As the "blank" fibre had low tenacity and extensibility, and as the calcium alginate A was of such poor quality, an attempt was made to purify it as follows—

1.5 kg. was leached free from calcium and left standing in *N.* hydrochloric acid overnight. The following morning a further acid treatment was followed by thorough water washing till free from chlorides. The alginic acid thus obtained was then dissolved in approx. 50 gal. of softened water, containing 880 g. of crystalline ( $\equiv$  326 g. of anhydrous) sodium carbonate, 100 c.c. of sodium hypochlorite solution (8.0% available chlorine) was added, and after an hour 11 g. of sodium metabisulphite as antichlor, together with 20 c.c. of 40% formaldehyde. The liquor was then allowed to stand for 48 hr. to settle the "weed residue". Precipitation of the decanted liquor was effected by running through glass tubing (6 mm. bore) into 22 litres of tap water containing 2 kg. of calcium chloride and 1 litre of concentrated hydrochloric acid, with continual stirring. The pH of the liquor at the end of the precipitation was 1.8; for ease of handling it was raised to 2.5 by the addition of 150 g. of calcium hydroxide. After standing for about an hour, the alginate was filtered off on canvas, and dewatered by hand as far as possible. It was then suspended in 10 litres of distilled water containing 100 g. of calcium acetate, milk of lime was added until the pH of the suspension was constant at 7.0, and the resultant calcium alginate was filtered off, washed with distilled water, and dewatered by hand. It was then rubbed through a sieve and air-dried.

A series of fibres (No. 9-14) was produced from this purified calcium alginate (for analysis, see Table I (B)). The calcium alginate "blank" (No. 9) showed some improvement in tensile properties over the previous "blank" (No. 1), but the dry extensibility was still much lower than was normally obtained.

In the next series (No. 15-18) the spinning dope was given an additional filtration prior to the candle filter. The effect of this is shown in the improved and normal dry extensibility of the blank (No. 15).

At this stage a supply of fairly good-quality *Laminaria* alginate became available, and to obviate the labour of cleaning up large quantities of the *Ascophyllum* alginate (A), it was decided to change over to this material. A 30-lb. sample was thoroughly mixed and analysed, with the results given under C in Table I.

A series of calcium-cadmium fibres (No. 20-24) was then produced from alginate C without initial filtration of the spinning dope. Again the blank calcium alginate fibre (No. 19) showed a poor dry extensibility. It was therefore decided to adopt as standard procedure the initial filtration of the spinning dope, and to assist this the alginic acid was degraded during the leaching stage for 1 hr. at 50°C.

This material was used for all subsequent experiments.

#### METHOD OF PREPARATION OF THE SPINNING DOPE AND SPINNING CONDITIONS

##### (a) Preparation of the Dope

The dope was normally of 5% concentration and was made up in 4-kg. batches. A quantity of the alginate containing 200 g. of calcium alginate was leached with successive portions of approx. *N.* hydrochloric acid on a No. 2 grade sintered-glass filter until the drainings were shown by the oxalate test to be free from calcium. The material was then washed with successive portions of distilled water until the filtrate was free from chloride when tested with nitric acid and silver nitrate. The above procedure was carried out at room temperature and occupied about 4 hr.

The alginic acid so formed was then transferred to a 2-litre beaker, and the total volume made up to approx. 1700 c.c. with distilled water at 60°C. The beaker and contents, which were continually stirred, were maintained at 50°C. for 1 hr. on the water-bath. The degraded alginic acid was transferred back to the sintered-glass filter and given two distilled-water washes at room temperature.

Dissolution of the alginic acid was effected by stirring with 2.5 litres of distilled water in a 5-litre jar until any lumps of acid had been broken up, when a slight deficiency of 10% aq. caustic soda was added and 2 c.c. of 40% formaldehyde to prevent microbiological degradation. The volume was made up to 4 litres, and the dope stirred overnight. The following morning the pH was adjusted to approx. 8 by the addition with stirring of further caustic soda solution.

The dope was then filtered by vacuum using a 10-in. Büchner funnel and a 5-litre flask. The filter material consisted—starting from the

TABLE I  
Analyses of Calcium Alginate

Batch ...	A	B	C
Origin ...	<i>Ascophyllum</i>	<i>Ascophyllum</i>	<i>Laminaria</i>
Moisture content, %...	42.1	43.6	34.6
Analyses			
(% on dry wt.)			
Calcium alginate ...	69.0	—	92.0
Weed residue ...	0.87	0.63	1.2
Water-soluble material ...	3.6	—	2.6
Acidity			
(as alginic acid, mol. wt. 194) ...	Nil	—	1.6
Nitrogen content (as N) ...	0.21	0.19	0.27
Sulphur content (as SO <sub>3</sub> ) ...	1.20	0.38	0.96
Viscosity			
(1% Na alginate soln.), centistokes	22	27	94

bottom—of  $\frac{1}{2}$ -in. mesh stainless-steel gauze to give drainage space, one layer of fine canvas, and four layers of coarse canvas. At the conclusion of the filtration the flask was left under vacuum, usually overnight, to remove air bubbles, and the dope was then ready to spin.

#### (b) Spinning Conditions

The spinning machine was made up from conventional viscose machine parts. The dope was fed by gravity from a reservoir to a gear pump, and thence to a candle filter dressed with fine canvas. A further filter of two layers of coarse canvas was placed behind the single-hole jet.

The jet was immersed in the coagulation bath, from which the thread was led via a guide to a godet and thence to a take-up reel. Gearing was arranged so that the pump output, the godet speed, and the take-up speed could be varied independently. The machine was set to spin approx. 150-denier monofil fibre.

Except in two early series (No. 1-5 (Table III): 7% dope concn. and increased godet speed; and No. 15-18: 7% dope) the spinning conditions were as indicated in Table II. Spinning was carried out under these conditions for approx. 40 min., yielding skeins of fibre weighing 30-35 g.

TABLE II  
Spinning Conditions

Dope concn. ...	...	5%
Output of pump, g./min. ...	...	16.1
Jet diameter, mm. ...	...	0.91
Immersion, cm. ...	...	115
Spinning bath—		
Calcium chloride ...	...	2%
Calcium acetate ...	...	2%
Acetic acid ...	...	0.25%
Sodium (as NaCl) ...	...	0.2%
Temperature ...	...	20°C.
Godet speed, metres/min. ...	...	59.3
Reel speed, metres/min. ...	...	61.7

Unless otherwise stated, the hank was then treated in three successive 2-litre baths containing 2% calcium chloride and 2% calcium acetate, the duration of each treatment being 20 min. If necessary, the pH of the final bath was adjusted to 7.0-7.5 by addition of lime-water. Previous work had indicated that this treatment effected practically complete removal of sodium from the fibre.

To produce a calcium fibre the hank was then given two distilled-water washes, the first of 30 min. and the second of 90 min. duration.

To produce a calcium-other metal fibre, the hank, after the first water wash, was given the appropriate treatment as indicated in Table III, followed by two distilled-water washes of 30 and 90 min. duration.

The hanks were cut, and the tresses of fibre hung up by one end to drain for 2-3 hr. They were then transferred to a well ventilated drying cupboard maintained at 35°C., where drying took place with the fibre under the tension due to its own weight.

When dry, the fibre was conditioned for at least 48 hr. at 21°C. and 65% R.H. before being tested.

#### AFTERTREATMENT OF THE CALCIUM ALGINATE FIBRE

##### Blank Calcium Alginate Fibre

In view of the influence which the quality of the calcium alginate has on the properties of the resultant fibre, a blank calcium alginate fibre was made, as described above, for each change in the calcium alginate or spinning conditions. In the case of calcium alginate C using a 5% degraded and filtered spinning dope, a number of calcium alginate blanks were made and tested. The results agreed within experimental error, and the average figures have therefore been used for comparison with those of the other metal-alginate fibres.

##### Calcium-Aluminium Fibres, No. 2-8

It is known that if an alginate fibre is immersed in a solution containing metal ions, an equilibrium is set up between the metal ions in solution and those combined with the alginate. It is also known that, if the solution is acid, a further equilibrium is set up resulting in the formation of a proportion of alginic acid in the fibre. The properties of samples No. 2-4 demonstrate this point.

To overcome this complication, which is inevitable with salts of weak bases and which would make it difficult to assess the value of the metal, the fibre, after treatment with aluminium sulphate solution, was suspended in distilled water, and lime-water added to neutralise the free alginic acid (sample 5). This took some time, as the uptake of calcium was very slow owing to the low concentration of calcium ions in the liquor.

In order to speed up this process, samples 7 and 8 were suspended in 0.1% calcium acetate and neutralised.

##### Calcium-Chromium Fibres, No. 10-14 and 44-61

During the development of calcium alginate scrim, considerable trouble was experienced with excessive shrinkage of the material on repeated wetting and drying. Speakman<sup>6</sup> suggested that this might be overcome by replacement of part of the calcium by basic chromium, and recommended the following procedure—

A solution of 31.8 g. of anhydrous sodium carbonate in 400 c.c. of distilled water is added gradually to a solution of 200 g. of chrome alum in 1500 c.c. of cold distilled water with constant stirring, and when effervescence of carbon dioxide has subsided, the mixture is made up to 2000 c.c.

The stock solution thus prepared is diluted five times before use, i.e. 200 c.c. of stock solution is diluted to 1000 c.c. (i.e. 0.2% Cr). The samples are treated in this diluted solution for 7 hr. at room temperature, with occasional stirring, using a liquor : fabric ratio of 30 : 1. After treatment, the samples are washed in running tap-water overnight, hydroextracted, and dried at room temperature under no tension.

Although the suggested treatment was recommended for the finished fabric, it was recognised that there would be advantages in producing a chromium fibre which could then be woven, etc. As the recommended procedure appeared to be open to objection in that the presence of sodium and potassium ions in the solution would lead to the formation of a proportion of sodium and potassium alginates, and the very long time of treatment

TABLE III  
 Properties of Alginate Fibres

No.	Batch	Calcium Alginate Concn. (%)	$\eta$ (1%, 25°C.)	Treatment (Ac = CH <sub>3</sub> CO)	Fibre Analysis (% on dry wt.)			Denier	Water Imbibition (%)	Water (%)	Tenacity (g./denier)		Extensibility (%)	
					Ca	Al	Alginic Acid				Dry	Wet	Dry	Wet
1	A	7	207	Ca alginate blank	9.21	Nil	Nil	118	209	22.2	0.53	0.17	2.1	3.5
				Fully calcified fibre immersed in 0.1 N-Al <sub>2</sub> (SO <sub>4</sub> ) <sub>3</sub> for—										
2				5 min.	2.57	4.31	40.5	118	140	22.0	0.59	0.20	0.77	5.0
3				10 min.	2.64	3.78	33.8	112	144	21.0	0.72	0.27	1.0	5.4
4				15 min.	1.86	3.89	46.0	98	154	22.1	0.88	0.32	0.66	4.3
5				Immersed in 0.2 N-Al <sub>2</sub> (SO <sub>4</sub> ) <sub>3</sub> for 20 min.; then suspended in water and neutralised with lime	5.98	3.45	8.95	125	100	23.6	0.58	0.24	1.1	20.0
6	C	5	85	Ca alginate blank	9.50	—	—	144	141	22.7	1.03	0.33	8.7	74.5
				Fully calcified fibre immersed for 2 hr. in—										
7				0.3 N-Al <sub>2</sub> (SO <sub>4</sub> ) <sub>3</sub>	7.4	3.4	—	182	95.5	24.0	0.77	0.50	10.6	45
8				0.1 N-Al <sub>2</sub> (SO <sub>4</sub> ) <sub>3</sub> then washed, suspended in 0.1% Ca(OAc) <sub>2</sub> , and free acid neutralised with lime	8.3	1.4	—	173	121.5	24.0	0.87	0.35	9.0	63
					Cr	K	Na							
9	B	5	20	Ca alginate blank	8.72	—	0.2	148	153	23.0	0.80	0.20	3.6	62
				Fully calcified fibre immersed for 2 hr. in—										
10				0.15 N-Cr(OAc) <sub>3</sub> + 0.067 N-Ca(OAc) <sub>2</sub>	9.15	0.18	1.5	148	162	23.0	0.80	0.24	4.3	37
11				0.15 N-Cr(OAc) <sub>3</sub> + 0.055 N-Ca(OAc) <sub>2</sub> + 0.015 N-Ca(OH) <sub>2</sub>	10.10	0.30	1.7	154	142	22.5	0.70	0.25	3.6	41
12				0.15 N-Cr(OAc) <sub>3</sub> + 0.044 N-Ca(OAc) <sub>2</sub> + 0.03 N-Ca(OH) <sub>2</sub>	8.72	0.85	0.8	150	143	22.5	0.77	0.24	3.8	36
13				Speckman's basic chrome alum (2.0 g. Cr per litre)	4.15	6.58	7.5	185	75	22.0	0.40	0.34	4.0	9.4
14				Cr(OAc) <sub>3</sub> (2.0 g. Cr per litre)	8.35	1.64	5.2	169	159	22.0	0.80	0.29	6.7	30
					Cu									
15	B	7	25	Ca alginate blank	10.1	—	1.1	210	143	21.0	1.10	0.23	11.6	54.0
				Fully calcified fibre immersed for 2 hr. in 0.2 N-Ca(OAc) <sub>2</sub> plus—										
16	Filtered			0.3 N-Cu(OAc) <sub>2</sub>	4.65	8.7	1.7	235	72.5	20.0	1.06	0.44	2.7	32
17				0.2 N-Cu(OAc) <sub>2</sub>	5.14	7.42	1.6	208	77.5	20.0	1.30	0.44	4.0	35
18				0.1 N-Cu(OAc) <sub>2</sub>	6.85	5.67	1.0	237	83.5	20.0	1.10	0.42	3.5	38.5

TABLE III (contd.)

No.	Batch	Calcium Alginate Concn. (%)	$\eta$ (1%, 25°C.)	Treatment (Ac = CH <sub>3</sub> CO)	Fibre Analysis (% on dry wt.)			Denier	Water Imbibi- tion (%)	Water (%)	Tenacity (g./denier)		Extensibility (%)	
					Ca	Cd	Alginic Acid				Dry	Wet	Dry	Wet
19	C	5	190	Ca alginate blank Fully calcified fibre immersed for 2 hr. in 0.2 N-Ca(OAc) <sub>2</sub> , plus— 0.3 N-Cd(OAc) <sub>2</sub> 0.2 N-Cd(OAc) <sub>2</sub> 0.1 N-Cd(OAc) <sub>2</sub> 0.05 N-Cd(OAc) <sub>2</sub> 0.025 N-Cd(OAc) <sub>2</sub>	9.71	—	Nil	152	139	22.6	1.00	0.25	5.3	60.5
20					5.58	5.86	Nil	157	94.5	19.9	1.06	0.45	5.9	33.5
21					6.35	4.90	Nil	171	95	20.1	0.84	0.35	2.8	37.5
22					6.93	3.94	Nil	169	102	20.8	0.87	0.39	3.2	36
23					7.65	3.15	Nil	156	115	21.0	0.87	0.39	3.3	39.5
24					8.72	1.66	Nil	157	118	21.5	0.94	0.42	3.7	53
25	C	5	85	Ca alginate blank Fibre spun in usual Ca bath, and finished in three successive 20-min. washes of— 0.2 N-Cd(OAc) <sub>2</sub> 0.3 N-Cd(OAc) <sub>2</sub> + 0.2 N-Ca(OAc) <sub>2</sub> 0.2 N-Cd(OAc) <sub>2</sub> + 0.2 N-Ca(OAc) <sub>2</sub> 0.1 N-Cd(OAc) <sub>2</sub> + 0.2 N-Ca(OAc) <sub>2</sub> 0.05 N-Cd(OAc) <sub>2</sub> + 0.2 N-Ca(OAc) <sub>2</sub>	9.50	—	Nil	144	141	22.7	1.03	0.33	8.7	74.5
26					0.57	20.2	Nil	180	79	19.2	0.82	0.46	8.2	51
27					5.00	12.35	Nil	177	95	21.0	0.86	0.45	7.5	56
28					6.00	10.50	Nil	185	96	20.1	0.95	0.45	8.6	51
29					7.00	6.40	Nil	169	102	21.2	0.98	0.42	9.1	45.5
30					8.50	4.82	Nil	161	107	21.9	1.04	0.48	10.5	53
31	C	5	85	Fully calcified fibre immersed for 2 hr. in 1.9 N-SnCl <sub>4</sub> , washed, suspended in water, and free acid neutralised with lime	6.29	11.80	4.6	232	84.5	17.7	0.37	0.14	3.6	26.5
32	C	5	85	Fully calcified fibre immersed for 2 hr. in 3.2 N-Bi(NO <sub>3</sub> ) <sub>3</sub> , washed, suspended in water, and free acid neutralised with lime	6.15	16.78	Nil	224	86	19.9	0.61	0.31	3.9	33
33	C	5	89	Fully calcified fibres immersed for 2 hr. in— 0.5 N-Ba(OAc) <sub>2</sub> 0.3 N-Ba(OAc) <sub>2</sub> + 0.2 N-Ca(OAc) <sub>2</sub> 0.2 N-Ba(OAc) <sub>2</sub> + 0.2 N-Ca(OAc) <sub>2</sub> 0.1 N-Ba(OAc) <sub>2</sub> + 0.2 N-Ca(OAc) <sub>2</sub> 0.05 N-Ba(OAc) <sub>2</sub> + 0.2 N-Ca(OAc) <sub>2</sub>	0.07	25.60	Nil	197	71.5	18.4	0.74	0.38	10.1	44
34					2.14	20.80	Nil	180	88.5	19.3	0.88	0.32	7.5	44
35					2.72	19.50	Nil	184	90.5	19.2	0.80	0.36	7.1	47
36					4.57	15.85	Nil	178	98.5	20.4	0.81	0.35	6.2	48
37	C	5	85	Fully calcified fibre immersed for 2 hr. in 0.2 N-Ca(OAc) <sub>2</sub> , plus— 0.3 N-Zn(OAc) <sub>2</sub> 0.1 N-Zn(OAc) <sub>2</sub>	5.79	13.45	Nil	175	102	20.4	0.89	0.28	7.4	51
38					0.71	11.89	1.1	162	121	23.0	1.10	0.31	8.8	57
39	C	5	85	Fully calcified fibre immersed <sup>a</sup> for 48 hr. in Be(OAc) <sub>2</sub> , and then— Boiling water-bath for 1 hr. Fibre washed and dried Fibre suspended in water and neutralised with lime. Washed and dried	0.71	10.08	Nil	162	135	23.0	0.91	0.33	9.1	61
40					3.00	4.54	0.4	152	49	22.4	—	0.55	—	4.3
41					4.00	4.21	0.4	159	52.5	23.2	0.97	0.72	3.3	5.3

TABLE III (contd.)

No.	Batch	Calcium Alginate Concn. (%)	$\eta$ (1%, 25°C.)	Treatment (Ac = CH <sub>3</sub> CO)	Ca	Be	Fibre Analysis (% on dry wt.) Alginic Acid	Denier	Water Imbi- tion (%)	Water (%)	Tenacity (g./denier) Dry Wet	Extensibility (%) Dry Wet
42				Fully calcified fibre immersed for 2 hr. in a more basic Be acetate soln. than for No. 40 and 41, and then— Boiling water-bath for 1 hr. Fibre washed and dried	1.86	5.80	0.4	200	48	23.8	—	—
43				Fibre suspended in water and neutralised with lime. Washed and dried	4.51	4.68	0.4	197	53.5	23.4	0.76	0.70 3.2 5.0
C	5	85		Dried and conditioned Ca alginate fibre was treated as shown below. It was then given two 30-min. distilled-water washes, dried, and conditioned. Speckman's basic chrome alum— Cr Temp. Time (%) (°C.) (min.)	Cr							
44				0.2 20 10	5.85	2.1	3.2	148	119	21.0	0.94	0.30 4.7 10.8
45				0.2 20 30	5.9	1.8	5.4	147	105	21.0	0.72	0.35 3.5 11.2
46				0.2 20 60	5.9	2.0	6.15	145	103	21.0	0.96	0.36 4.7 10.1
47				0.2 20 120	5.4	3.0	6.1	151	89.5	21.8	0.87	0.32 3.8 9.3
48				1.04 20 20	2.1	8.2	11.7	190	74	21.9	0.72	0.25 3.5 5.7
49				0.78 20 20	3.1	4.95	9.55	172	77	22.0	0.82	0.28 4.9 6.0
50				0.50 20 20	5.2	4.1	7.15	169	93	22.9	0.63	0.20 4.3 5.1
51				0.26 20 20	6.65	2.5	5.25	183	101	22.4	0.81	0.29 6.9 12.0
52				0.17 20 20	6.8	2.25	3.1	152	119	21.9	0.94	0.30 6.3 14.1
53				0.2 20 20	5.9	1.85	4.8	149	130	21.5	0.88	0.25 3.8 9.0
54				0.2 35 20	5.6	3.3	7.2	170	132	20.5	0.85	0.32 4.8 11.7
55				0.2 50 20	6.5	3.4	9.25	148	149	20.5	0.96	0.21 5.1 9.7
56				0.2 75 20	5.65	2.6	14.25	151	193	20.5	0.83	0.045 4.8 7.6
57				0.2 100 20	5.45	1.3	22.0	141	198	19.5	0.73	0.033 2.3 8.6
58				Chromic alum soln. (0.2% Cr) plus Na <sub>2</sub> CO <sub>3</sub> equivalent to (20 min. at 20°C.)— 0% Cr	7.25	3.15	15.25	147	83	21.0	1.02	0.41 3.6 9.6
59				0.05% Cr	6.65	2.8	11.8	147	94	21.3	0.83	0.40 3.0 7.7
60				0.1% Cr	7.35	1.8	6.7	144	120	21.0	1.08	0.42 5.8 13.0
61				0.132% Cr	7.4	1.2	3.75	143	161	21.5	1.03	0.27 5.0 13.6

(7 hr.) would introduce difficulties in large-scale production, the following experiments were carried out.

**DIRECT PRODUCTION OF CALCIUM-CHROMIUM FIBRE**—Samples 10–12 were treated for 2 hr. in solutions containing 0.3% chromium and 0.15% calcium which had been made increasingly basic by the use of calcium hydroxide instead of the equivalent amount of calcium acetate.

Sample 13 was made by using Speakman's basic chrome alum solution but with an immersion time of 2 hr. only, while No. 14 used chromium triacetate only.

**AFTERTREATMENT OF THE DRY CALCIUM ALGINATE FIBRE**—This series of samples (No. 44–61) was prepared under conditions designed to show the influence of the time of treatment (44–47), concentration (48–52), temperature (53–57), and basicity (58–61) on the uptake of chromium, but with a much shorter time of treatment than the recommended 7 hr.

#### *Calcium-Copper Fibres, No. 16–18*

These were produced in the normal manner as described above.

#### *Calcium-Cadmium Fibres, No. 20–24 and 26–30*

No. 20–24 were produced from fully calcified fibre, whilst No. 26–30 were made from freshly spun fibre which still contained some unconverted sodium alginate.

#### *Calcium-Tin and Calcium-Bismuth Fibres, No. 31 and 32*

Both stannous chloride and bismuth nitrate yield acid solutions, and in both cases a small quantity of hydrochloric and nitric acids respectively had to be added to the solutions to prevent precipitation of basic salts. The resultant fibres were therefore strongly acid, and were afterwards suspended in 0.1% calcium acetate and neutralised with lime-water.

#### *Calcium-Barium Fibres, No. 33–37*

These were produced in the normal way, with the added precaution that all the water used was boiled and cooled to prevent precipitation of barium carbonate on the fibre.

#### *Calcium-Zinc Fibres, No. 38 and 39*

These were made in the usual way and required slight neutralisation after treatment.

#### *Calcium-Beryllium Fibres, No. 40–43*

These were produced by the technique described by Speakman and Chamberlain<sup>5</sup>.

The experimental details are summarised in Table III.

### **Fibre Testing**

#### **DENIER**

The denier was determined by measuring 9 metres of the fibre, drying for 4 hr. at 100–105°C., and weighing, it having first been determined that fibres of this type reach constant weight under these conditions. The moisture in the conditioned yarn is recorded as “% moisture content” and not as “% moisture regain”.

#### **WATER IMBIBITION**

A 5-g. sample of the fibre is given three half-hour washes each with 300 c.c. of boiled distilled water at 20°C., the sample being allowed to drain for 5 min. between each wash. It is then freed from surplus water by repeated pressing between several layers of filter paper until no moisture mark is obtained on the paper. The sample is then weighed, dried for 4 hr. at 100°C., and reweighed.

$$\text{Water imbibition (\%)} = \frac{(\text{Wet weight} - \text{Dry weight}) \times 100}{\text{Dry weight}}$$

This method possesses the advantage that the moisture content of the sample need not be known, and obviates any error due to the presence of water-soluble salts in the fibre.

#### **DRY AND WET TENACITY AND EXTENSIBILITY**

These were determined by means of a Goodbrand single-thread tester by the procedure laid down by the B.I.S.F.A.<sup>7</sup>

The “dry” tenacities found for the calcium and beryllium alginate yarns are low compared with those recorded by Chamberlain, Johnson, and Speakman<sup>6</sup>. This is due to the fact that these workers used fine yarns having filament deniers in the range 2–5, whereas in our investigation coarser monofilaments in the denier range 100–200 were used.

The results of these tests together with the summarised preparative details are recorded in Table III.

### **Discussion and Conclusions**

A consideration of the tabulated results leads to the following conclusions with regard to the properties of calcium alginate fibres modified by partial replacement of calcium by the metals examined.

#### **ALUMINIUM**

Providing that the fibre is neutral, the substitution of calcium by aluminium has a beneficial effect. There is some loss in dry tenacity, but the wet tenacity is increased and there is only a moderate loss in wet extensibility. The water imbibition is reduced to a satisfactory extent. The only drawback is the necessity for neutralising the fibre after the aluminium treatment.

#### **BARIUM**

Barium is comparable with cadmium, although the increase in wet tenacity is not quite so great. The main drawback to producing barium alginate fibre is the necessity to eliminate sulphates and carbonates from the baths and wash waters to prevent precipitation of barium sulphate or carbonate, although this could probably be overcome by means of ion-exchange processes.

#### **BERYLLIUM**

All these fibres are extremely brittle. The boiling treatment of Speakman exaggerates this to such an extent that it was impossible to obtain the dry tenacity and extensibility figures, and in one case the wet tenacity also. This can be attributed to the high beryllium content, which has reduced the water imbibition below that obtained with any of

the other metals which have been tried. The low water imbibition is also reflected in the very low wet extensibility. It is possible that small proportions of beryllium would give adequate reduction in water imbibition and still maintain a reasonable extensibility. The fibre is noteworthy for the high ratio of wet to dry tenacity.

#### BISMUTH AND TIN

Apart from the complication due to the acidity of stannous and bismuth salt solutions, which necessitates the addition of a considerable quantity of lime to neutralise the alginic acid formed, the resultant fibres both show inferior textile properties to those of calcium alginate.

#### CADMIUM

Substitution of calcium by cadmium has given an all-round improvement in properties without any disadvantages. With this metal there is a lowering of water imbibition and an increase in wet strength without the loss in dry extensibility which has occurred with most of the other metals.

#### CHROMIUM

##### *Direct*

Chromium contents up to 1.6% chromium on the dry fibre have little effect on the properties of the fibre apart from some lowering of wet extensibility. Sample 13, with a chromium content of 6.58%, shows only half the water imbibition of the calcium blank, and the wet tenacity has been increased by 60%, but the dry tenacity is halved and the wet extensibility reduced to one-sixth.

##### *Aftertreatment*

As there are varying amounts of alginic acid in these fibres, it is difficult to draw reliable conclusions. In general, increased chromium content leads to a decrease in water imbibition, although aftertreatment above 50°C. gave high values.

There is some loss in dry tenacity and little change in wet tenacity except in the cases of the high-temperature and the less basic samples. In all cases there is a decrease in dry extensibility and a considerable loss in wet extensibility.

The greenish-violet colour common to all samples deepens with increased chromium content.

#### COPPER

The presence of copper lowers the water imbibition and increases the wet tenacity at the expense

of the dry extensibility. There is also a reduction in wet extensibility. The blue colour of the fibre is a drawback.

#### ZINC

Despite almost complete replacement of calcium, only slight reduction in water imbibition and no loss in tensile properties resulted.

In the case of all the eight metals examined, a reduction in water imbibition was found, the magnitude of the reduction increasing with the amount of calcium replaced. With the exception of aluminium, barium, and cadmium, however, this was accompanied by a reduction in dry and wet extensibilities, i.e. the fibre becomes more brittle.

Of these three, cadmium shows most promise, since, compared with calcium alginate fibre, it shows no loss in dry extensibility, it has an increased wet tenacity, and only a small loss in dry tenacity. Moreover, unlike aluminium- and barium-modified fibres, it can be produced with no more difficulty than calcium alginate.

It is conceivable, although it has not been proved, that a combination of the desired characteristics would be obtained in yarns made from beryllium-cadmium alginate.

\* \* \*

This investigation was carried out during 1943 at the Ministry of Home Security (later Ministry of Supply) Research Establishment, Leamington Spa. Acknowledgment is made to the Chief Scientist, Ministry of Supply, for permission to publish and to Mrs. M. D. Eastham for undertaking the chemical analyses. Our thanks are due also to Dr. N. H. Chamberlain for helpful suggestions in the preparation of this paper.

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(Received 18th September 1953)

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## CORRESPONDENCE

*The Editor does not hold himself responsible for opinions expressed by correspondents*

### The Effect of the Soaping Aftertreatment on Vat Dyes

I have read with interest Messrs. Sumner and Vickerstaff's letter<sup>1</sup> to you on the above subject.

I am pleased to see that the sentiments expressed in their letter, and the findings which they published in their paper last year<sup>2</sup>, fully agree with the results of my work, published in this *Journal* in 1942<sup>3</sup>, in which it was clearly demonstrated that the colour change which vat and azoic dyeings undergo on soaping is due to transition of the dye from the amorphous to the crystalline state.

When Dr. Wegmann's paper appeared<sup>4</sup>, I pointed out to him in private correspondence that insufficient reference was made to my work, and also that his findings in no way contradicted the opinions expressed in my paper. Unfortunately, this private correspondence remained rather one-sided, and I felt, therefore, the need of adding a final paragraph to my contribution to this *Journal* concerning *Colour Changes induced in Vat Dyes by Organic Solvents*<sup>5</sup> that the crystallisation which is thought to underlie the colour change need not necessarily lead to the formation of microscopic crystals, but that rearrangement of the dye aggregates in the form of an orderly crystal lattice is quite sufficient to effect the colour change.

I should, therefore, like fully to support Messrs. Sumner and Vickerstaff's argument that there is no need to introduce a new theory into an issue which seems to be already cleared up.

E. KORNREICH

63 DOLLIS PARK  
LONDON N.3  
2nd April 1954

<sup>1</sup> Sumner and Vickerstaff, *J.S.D.C.*, **70**, 123 (1954).

<sup>2</sup> Sumner, Vickerstaff, and Waters, *J.S.D.C.*, **69**, 181-194 (1953).

<sup>3</sup> Kornreich, *J.S.D.C.*, **58**, 177 (1942).

<sup>4</sup> Wegmann, *Textil-Rund.*, **8**, 4-9, 97-108, 157-165 (1953).

<sup>5</sup> Kornreich, *J.S.D.C.*, **69**, 297 (1953).

### Development of the Geometric Grey Scale

The Color Committee of the American Association of Textile Chemists and Colorists has been asked by the latter's research heads to comment on the discussion by the Society's Fastness Tests Co-ordinating Committee (F.T.C.C.) of the history of "The Development of the Geometric Grey Scales for Fastness Assessment"<sup>1</sup>. The Chairman of the A.A.T.C.C. Color Committee is happy to have received an advance copy of the very lucid account of the history of the subject prepared by the F.T.C.C. Our committee, however, cannot say that the British position, as delineated in that article, is wholly a logical or felicitous one, and occasions considerable surprise.

The F.T.C.C. has attempted, with some success, to draw three major conclusions—

1. That a scale of properly stepped grey samples is adequate for visual judgment of the magnitude of fastness-test colour differences between chromatic (non-grey) samples, so that a

single number can be assigned to the size of the colour difference.

2. That the grey scale should have its steps geometrically spaced visually rather than arithmetically.

3. That better success attends the judgment of equality of "contrasts" (pairs of differing greys) than judgment of the positions on the grey scale of both "original" and "faded" samples, and noting the difference.

Certainly the first two and probably also the third point come as no surprise. When the undersigned—"undersigned" is used here several times to avoid the cumbersome phrase "chairman of the A.A.T.C.C. Color Committee"—set up the colours of the present Munsell system in the late 1920s, it had to be assumed that an evenly stepped grey scale, determined by elaborate and costly experiments, was valid for chromatic colours. Several of us then in the Munsell Laboratory found it possible to resort, with fair precision, to a "heterochromatic photometry", which included comparison of greys with chromatic colours. In the late 1920s the undersigned frequently asked observers to make exactly that sort of judgment which the F.T.C.C. now asks us to make.

That the most useful scale of magnitudes of test differences was visually geometric rather than arithmetical was clearly embraced in the undersigned's comments on the textile industry's usual scale of "trace", "noticeable", "appreciable", etc., difference quoted from a 1939 letter from him by Dr. D. B. Judd in his pioneering paper on "Specification of Uniform Color Tolerances for Textiles"<sup>2</sup>.

Concerning point three one needs to say only that if there is any validity in the Gestalt psychology, point three is almost to be expected. One may add that the overall precision of two independent judgments of visual position on a scale is very apt to be less than a single judgment of the magnitude of a "contrast" (colour difference in a pair).

We now turn to some obvious inconsistencies in the British point of view and to the unhappy state of their defeatist attitude in one important particular. For it is surely a defeatist attitude to denounce the use of *all* colour difference equations applied to objective measurements of fastness-test differences, because *two* among many equations or methods were found unsuccessful. These were the Adams method used by Vickerstaff, by Ricketts, and by the undersigned, and his "Newtonian" equation. The latter can be dismissed by saying that his chief object in its publication was to show that, *if* colour space is Newtonian, and one makes only the simple "straight-line mixture" assumption first made by the British physicists Newton and Maxwell, then one arrives at his published equation, which may or may not give a rough approximation to the truth about colour differences. But for the well known evidence that colour space is *not*

Newtonian, one may consult Dr. D. B. Judd's book<sup>3</sup>.

Concerning the Adams method, one may say that *colour science has not stood still*, and improved modifications of this method have been published (one by the undersigned<sup>4</sup>) since it was first applied to fastness tests. Since the publication of his two papers quoted by the F.T.C.C.<sup>5,6</sup>, he has studied the validity of several equations by means of visual judgments and measurements on three sets of colours totalling over 600 colour differences. Two or three of the equations proved better than the equation derived from the Adams method. This is not surprising, for the Adams method gives an egg-shaped constant-chroma locus for the Munsell colours (in the form of the "renotation" colours derived from over four million observations by a large committee of experienced observers). If the Adams method were entirely valid, the constant-chroma locus would be expected to be a circle. The points of greatest relative eccentricity are met when comparing yellow swatches with blues. In the undersigned's 1950 paper<sup>5</sup>, he applied the method only to the simpler case of comparison of faded blue samples with faded blue standards, where most errors may cancel.

Since publication of the British article<sup>1</sup>, however, the Adams method has received further approximate verification in a study by two du Pont colour workers, who obtained a 63% correlation coefficient when comparing an Adams-Nickerson colour-difference index with visual judgments of magnitude of colour change<sup>7</sup>. They, too, found the nature of the correlation somewhat different for yellows and for blues (and purples). Their data will be recomputed to see whether they yield an expected still better correlation when judged by the modified Adams method<sup>4</sup>, which yields a nearly circular constant-chroma locus instead of the egg-shaped one of the original Adams method.

Among other methods the "MacAdam ellipses" as a basis for a unit of colour measurement have been very successfully applied in this country by MacAdam, by Derby, by Davidson, and by Ingle. Until this method and the N.B.S. (National Bureau of Standards) method have been tried and found wanting (and perhaps similarly one or two other equations examined in the undersigned's extensive studies of over 600 colour differences), the F.T.C.C. is certainly not justified in saying<sup>8</sup> that it "*has had sufficient experience of colour-difference formulae to be convinced that their use in fastness testing is dangerous*". This statement is especially astonishing when coming from a committee whose members must all be so well aware of the fine pioneering work of P. W. Cunliffe in the application of objective measurements in the field of fastness testing.

How can the F.T.C.C. justify the categorical italicised statement rejecting *all* colour-differences equations and methods because two equations have failed—if indeed they have, especially when one of these has a known defect since remedied in the 1952 paper<sup>4</sup>, and the other has been discarded by its author, since derived almost wholly for a specific purpose foreign to the present problem? What

experience has the F.T.C.C. had with the "MacAdam ellipse" method which has been in successful practical use by three members of the A.A.T.C.C. Color Committee, and by at least two other industrial scientists?

Moreover, the F.T.C.C. must find itself in a very illogical position in failing to keep in mind the Hunter equations which define the N.B.S. (Judd) unit of colour difference. For in the same document in which they decry the use of *all* colour-difference equations, they make use several times of this unit defined by the Hunter equation. Incidentally, one statement is definitely incorrect: The N.B.S. unit is *not* a "just perceptible step". Judd has very clearly stated that it was intended to be about four or five times as large as a just perceptible step.

We turn now to the work of Ricketts<sup>9</sup>, which seems to have influenced the F.T.C.C. greatly. They state that Ricketts, using the Adams equation (as Godlove did), confirmed the Godlove finding that the British light fastness standard<sup>10</sup> 5 was not as fast as standard 4. But Ricketts is stated to have proved, by visual judgments, that standard 5 "is twice as fast as" standard 4, and that both the Adams formula and Godlove's Newtonian one give results contrary to visual observation.

Ricketts<sup>9</sup> has not previously been answered by the undersigned, because the measurements reported in the Godlove 1950 paper<sup>6</sup> were on already exposed samples delivered to him by the A.A.T.C.C. Light-fastness Committee. The undersigned could measure only what was delivered to him. Being confident that errors of several hundred per cent. or more could not possibly be present either in his spectrophotometric measurements or in the Adams method, he was forced to conclude either that the British light-fastness standards were far less uniform than the American, or that some accident had occurred in the exposures delivered to him for measurement. It was decided that in the time available far more important work than repetition of the experiments in question could be accomplished by exhaustive comparisons of visual judgments of colour differences with measurements plus computations from various equations. Thus resulted the studies referred to above of 600-odd visually calibrated samples by each of a number of equations.

We need not press a third possibility of source of error, namely the relatively large errors that can be made in visual judgments even by ten observers whose results are averaged by valid statistical procedures. The I.C.I. colourists have published examples in a related judgment, that of strength; the Chairman of the Color Committee has found it very easy to deceive the eye into departing far from objective reality by careful choice of samples, and he is sure that Dr. D. B. Judd could furnish many other examples. The latter could show the F.T.C.C. how seriously visual judgments are disturbed by varying sharpness of dividing line between compared samples and by texture differences. Mr. W. D. Appel, also of the National Bureau of Standards, has much evidence of the last-named effect.

Moreover, knowing that the Adams method is based on relations between colours studied with over 4,000,000 observations by many more than Ricketts' ten observers, the undersigned is reluctant to believe that the Adams method can possibly lead to errors of over 300% (especially in comparing only blues with blues, where errors tend to cancel, as he used the method), even though he is sure moderately large errors are present. (This is especially true, since he has recently applied it very successfully to measurements of "depth" or "apparent strength" of dyeing). It must also be recalled that Godlove's 1950 paper<sup>5</sup> found variations of 1000% (0.2 unit to 2 units) in the readings of the several British Standards, while the maximum variation in the American set was 300%. We are not talking here about errors of, say, only the 30 or 40% one might find in the Adams method.

It seems to the undersigned that it is only reasonable to expect larger variations in the British standards than in the American standards, because the British standards were prepared with several different dyes, while the American set were prepared in all cases with the same two dyes.

It possibly has some significance that Ricketts found his No. 4 grey standard the duldest of the six he studied, that is, the closest to a true grey, and this was the standard which at various exposure times gave greater agreement between visual observation and calculation. In view of the proved difficulties in judging colour differences between bright (saturated) samples, this result may be in part due to failure of the visual estimates on the standards other than No. 4. The same errors may occur in using the greys to judge other colour differences.

Again, the F.T.C.C. is in the peculiar position of decrying the use of the Adams equation, yet using it to define the N.B.S. unit. Further, the quoted formula is not the best one to define the number of N.B.S. units in any scale or colour difference, particularly a grey scale. It merely embodies Miss Nickerson's constants determined to give the best fit of the Adams equation to a certain body of experimental data. It defines an N.B.S. unit only in the sense that *on the average*, for the colours she studied, the number of N.B.S. units calculated by her (quoted) formula agrees with the number calculated by the N.B.S. (Hunter) equations. Her formula, accepted by the F.T.C.C., uses the constant 40; Judd uses 51 in his book, and the undersigned found 51.5 experimentally. When dealing with a strictly grey scale, the Adams-Nickerson equation reduces to a simple one which is *not* identical with the N.B.S. equation. Unless the British article is misunderstood, the F.T.C.C. has used the Adams-Nickerson equation, or a degenerate form of it, all through its article.

One cannot agree with the last paragraph of the document, that colour difference formulae have "inestimable value" for comparing grey scales, at least the non-selective greys. These formulae are intended to combine correctly a chromaticity (shade plus brightness) difference, which does not exist in comparing greys, with a difference in lightness (measured by reflectance). It is much more direct

to use the reflectance (Y-value) portion of the N.B.S. equations. One may be confident that the proper precautions in measurement and visual judgment will make 3 N.B.S. units of measured-and-calculated difference more nearly equal to 3 N.B.S. units of visual difference when using the grey scale for reference.

The undersigned agrees with the F.T.C.C. that all calculations must yield "what the eye sees". But both the data for translation and the visual judgments to be compared must be made under carefully controlled conditions, which only long experience dictates. For example, it is well known to all experienced in the field of colorimetry that visual judgments of lightness difference (especially in comparison to chromaticity difference) are very greatly influenced by the dividing line between samples compared and their degree of contiguity, as well as by texture and other variables.

Finally, while the Color Committee thinks that the F.T.C.C. has been a little uncharitable to the contributions from America, it does not quarrel with the latter's three main conclusions listed above. Ricketts' work, except for the possible criticisms mentioned above, is regarded as a valuable contribution. But the F.T.C.C. article contains a number of errors of fact and apparent misunderstanding which should be corrected to avoid misleading British and Continental colourists. Whether the chairman of the Color Committee was right or wrong in his quoted 1950-51 papers is much less important than the general questions discussed here, for example, whether objective measurement and computational translation to visual magnitudes is "dangerous" or valueless. We are happy to see the general directions in which the F.T.C.C. wishes to go and say "amen" to Cunliffe's suggestion of the use of "contrast" in the Grey Scale. The undersigned hopes to apply to fastness test data a new unpublished formula which is not as accurate as the N.B.S. equations, but enormously simpler. He is hoping also that MacAdam, Ingle, Davidson, or Derby in this country will apply the very promising "MacAdam ellipse" method to the same end. It is hoped, and certainly not to be wholly unexpected, that further progress will lead to better agreement between objective measurement and visual judgments in fastness testing, as it has in other colour fields in the hands of several workers in this country.

For the A.A.T.C.C. Color Committee,  
I. H. GODLOVE, *Chairman*

15th February 1954

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<sup>1</sup> Fastness Tests Co-ordinating Committee, J.S.D.C., 69 404 (Nov. 1953).

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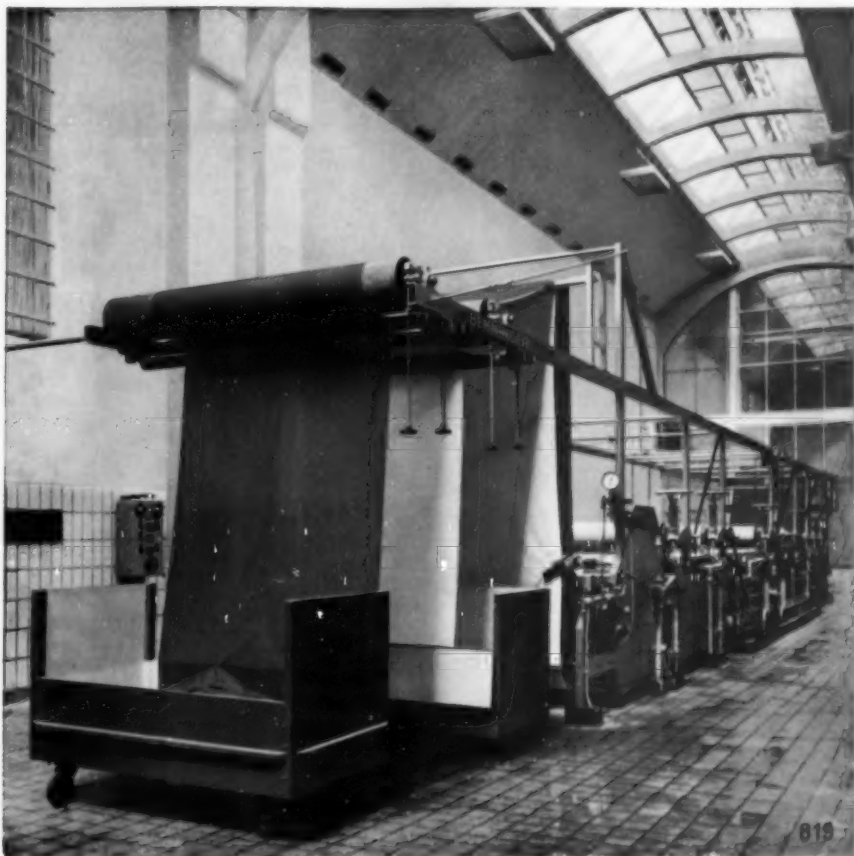
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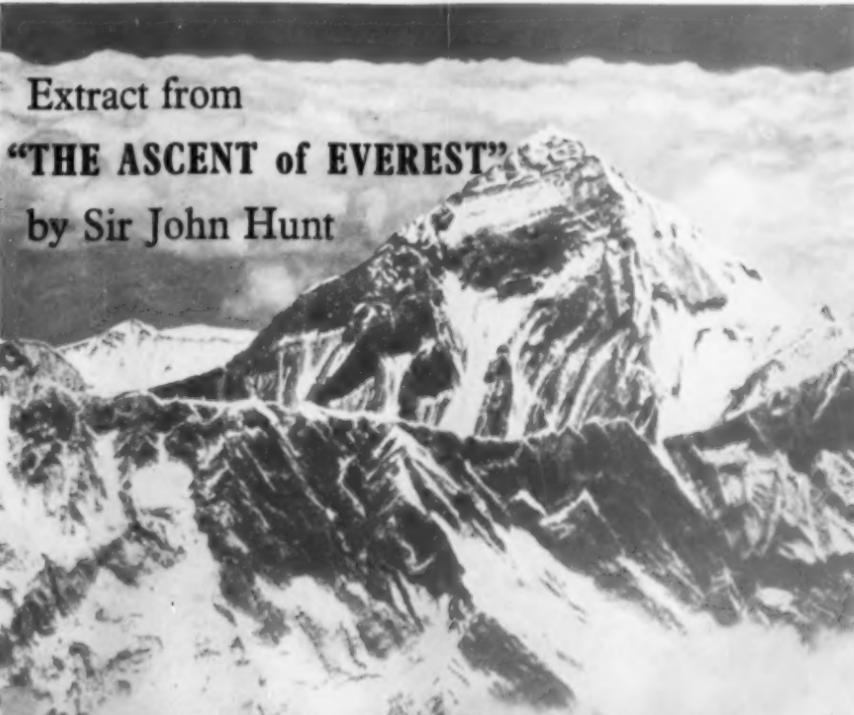
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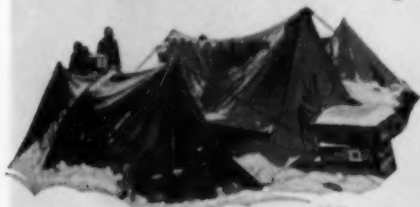
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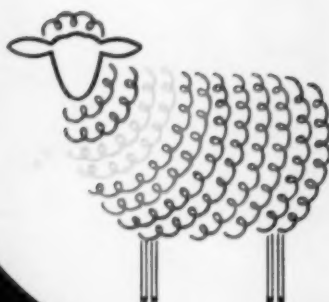
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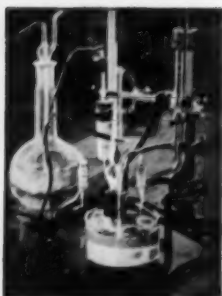
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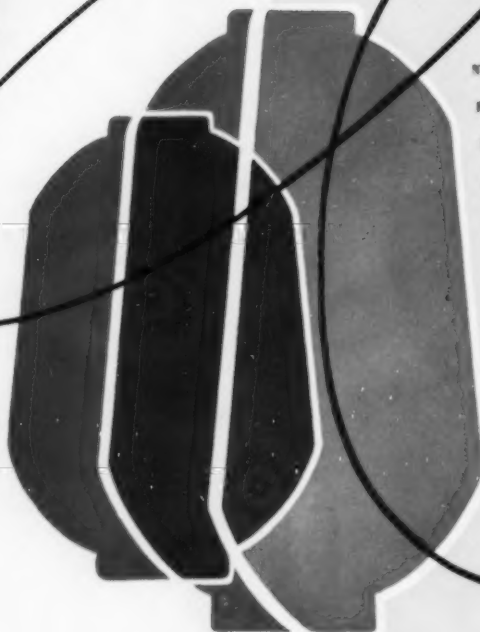
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<sup>7</sup> Webber, A. C., and Billmeyer, F. W., *ibid.*, 43, 1127 (1953).

<sup>8</sup> F.T.C.C., *loc. cit.*, p. 408.

<sup>9</sup> Ricketts, R. H., *J.S.D.C.*, 68, 200 (1952).

<sup>10</sup> B.S. 1006: 1953 (London: British Standards Institution).

The final section of the historical account of the development of the Grey Scales was intended to act as a warning against indiscriminate use of colour-difference formulae in fastness testing. Unfortunately, Dr. Godlove has read much more into this than was intended, and is under the mistaken impression that the F.T.C.C. reject outright all colour-difference equations. This is not true, but the Committee is of the opinion that too much reliance has been placed in the past on such methods in the U.S.A. In view of Dr. Godlove's letter, therefore, it would perhaps be best if the main evidence which led to this conclusion were presented in greater detail.

The first indication that colour-difference equations were unreliable in certain cases was the publication of the results of an investigation by Dr. Godlove<sup>1</sup> into the spacing of the U.S. and European light-fastness standards. The general conclusions reached were that the European standards, unlike the American, were very badly spaced.

The most surprising result was that the British Standard 5 was not as fast as Standard 4, whereas it was supposed to be twice as fast. If this anomaly were real—and the criterion of reality in this connection must be visual perception—then it would be incredible that it had not been detected earlier, either during the sixteen years these standards were in daily use in the United Kingdom, when Standards 4 and 5 must have been exposed together tens of thousands of times, or during the investigation which led to their adoption by the A.A.T.C.C. in 1937 as Tentative Standards.

The anomaly was subsequently resolved by Ricketts<sup>2</sup>, who found that the application of the two equations used by Godlove in 1950<sup>1</sup> and 1951<sup>3</sup> gave results completely different from average visual perception and that the British Standards were, in fact, spaced as intended, i.e. each being twice as fast as the preceding one.

Dr. Godlove admits that the Adams method may give errors of 30–40%; apparently he has not, however, realised that if in the case of Standard 4 the measured value is 35% too low, and for Standard 5 it is 35% too high, then Standard 4 will appear to be the faster.

The second anomaly appeared when the F.T.C.C. received examples of the official A.A.T.C.C. "just appreciable change in colour" prepared by exposing Standard I4 for 20 standard fading hours; Mr. Appel stated (and it was subsequently confirmed by the F.T.C.C. using the Adams method) that the measured colour difference was 2.4 N.B.S. units.

Subsequently, the A.A.T.C.C. light fastness Committee proposed that the Geometric Grey Scale (the spacing of which had been agreed between the F.T.C.C., the European fastness committees (E.C.E.), and the A.A.T.C.C.) should be altered from 0, 1.5, 3, 6, 12 N.B.S. units to

0, 1.25, 2.5, 5, 10 N.B.S. units, so that one contrast would have approximately the same measured value as the standard A.A.T.C.C. fade.

The F.T.C.C., however, thought that the standard fade was visually much less than a grey contrast of 2.4 N.B.S. units, but, appreciating that visual assessments show great variations, obtained the opinions of 40 experienced colourists. Their assessments are given as a frequency distribution in Fig. 1.

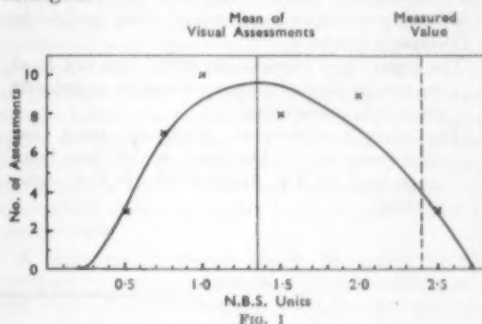


FIG. 1

Dr. Godlove mentions that relatively large errors can be made in visual judgments; in order to prove that this cannot explain the discrepancy between mean visual and measured assessments, the results of the 40 observers were subjected to elementary statistical analysis. This showed that the 95% Confidence Interval extended from 1.16 to 1.54 N.B.S. units, and as the measured value lies well outside these limits, it must be assumed that in this case the colour-difference equations used are responsible for the discrepancy.

The F.T.C.C. has sponsored several investigations into visual assessment<sup>2, 4, 5</sup> and is well aware of the variability even among experienced colourists. Consequently the Committee would welcome the use of colour-difference equations, providing their reliability had been completely established. Unfortunately, however, too many serious discrepancies have been observed, and the two quoted in detail above might have had serious consequences.

It would be interesting to know whether the application to these two cases of the new and improved colour-difference equations mentioned by Dr. Godlove would give values more in line with average visual assessment, and the F.T.C.C. would welcome information on this point.

The main purpose of the warning note was therefore to prevent the assumption being made that two contrasts which are equal by measurement would also be equal visually. There appeared to be a considerable danger that, if the 0, 1.5, 3, 6, 12 Grey Scale became internationally accepted (as well it might), then colour differences obtained by measurement might be converted into the corresponding 1–5 grade, e.g. a measured contrast of 1.5 N.B.S. units might be rated Grade 4 merely because Grade 4 has a measured contrast of 1.5 N.B.S. units. The F.T.C.C., perhaps, pays more attention to the failures of the various colour-difference formulae than do some American workers in this field as, if they do not correlate the measured with

the mean visual—and the above examples are obviously two cases where they do not—then clearly considerable argument over anomalies might result. The protagonists of the measurement method would, as Dr. Godlove himself has done, refer to the large variations in the assessments of experienced colourists; yet, in each of these cases, the measured value is far removed from average visual assessment.

The Committee would also like to make the following observations concerning other points in Dr. Godlove's letter—

- (1) The term "just perceptible step" was not used in the published paper except to point out that it is misleading.
- (2) The colour-difference formula used for measurement of the grey scales has been approved by Dr. Judd of the N.B.S., who states—

We have no objection to the use of this formula in spite of the fact that it differs somewhat from the original definition of the N.B.S. unit.

As there is no change in hue in the scales, the formula actually reduces to  $\Delta E = 9.2 V_y$ , which Dr. Godlove approves; the complete formula was quoted, however, as certain grey scales had been prepared experimentally which did show hue variations.

#### FASTNESS TESTS CO-ORDINATING COMMITTEE

31st March 1954

<sup>1</sup> Godlove, *Amer. Dyestuff Rep.*, **39**, P 215 (1950).

<sup>2</sup> Ricketts, *J.S.D.C.*, **68**, 200 (1952).

<sup>3</sup> Godlove, *J. Optical Soc. Amer.*, **41**, 763 (1951).

<sup>4</sup> Davies and Marney, *J.S.D.C.*, **67**, 438 (1951).

<sup>5</sup> McLaren, *ibid.*, **68**, 205 (1952).

## Notes

### Meetings of Council and Committees

#### April

Council—14th

Publications—21st

Perkin Centenary—8th

Colour Index Editorial Panel—27th

#### May

Council—12th

Publications—18th

Colour Index Editorial Panel—27th

Perkin Centenary—18th

Diploma Executive Subcommittee—12th

Fastness Tests Co-ordinating—21st

Terms and Definitions—1st

Mercer Lecture—21st

#### Death

We regret to report the loss by death of Mr. F. Wilkinson.

### Fourth Canadian Textile Seminar

Kingston, Ontario, 9–11th September 1954

Registration forms and full details of the Seminar, to be held at Queen's University, may be

obtained from the Textile Technical Federation of Canada, 1410 Guy Street, Montreal 25, Quebec, Canada. The following papers will probably be of special interest to readers of the *Journal*—

*The Sorption of Polar Substances by Textile Fibres* P. Larose

*The Effect of Sunlight on Cellulosic and other Textile Fibres* M. L. Staples

*The Soiling Characteristics of Textile Fibres* A. S. Weatherburn

*Control of Fabric Degradation in the Application of Thermosetting Resins* H. C. Borghetty

*New Developments in Pigment Printing on Textile Fibres and Novelty Screen Effects* F. G. LaPiana

*Principles of Textile Colour Control* R. E. Derby

*Scouring Raw Wool with Non-ionic Detergents and Salt* H. Zimmerman

*Research to transform Cotton through Chemistry into New Textile Products* C. H. Fisher

*The Dyeing of Wool and Wool-Synthetic Blends* H. D. Grimes

### International Technical Textile Conference

Barcelona, 4–9th October 1954

Papers are invited for the Conference, which includes a section devoted to bleaching, dyeing, printing, and finishing. It is being held to celebrate the fiftieth anniversary of the institution in Spain of the degree of "Textile Industries Engineer". Full details may be obtained from the Asociación Nacional de Ingenieros de Industrias Textiles, calle Mallorca 279, Barcelona, Spain.

## New Books and Publications

### Dictionary of Organic Compounds

Editors-in-chief Sir Ian Heilbron and H. M. Bunbury. New four volume edition 1953. Pp. (1) xvi + 654, (2) xvi + 845, (3) xvi + 838, (4) xvi + 694. London: Eyre & Spottiswoode. Price, each volume, £7 net.

A sense of keen satisfaction will pass through the organic chemical world when it is realised that once again this dictionary is available, and in as up-to-date a condition as human frailty and ingenuity can make it. When the dictionary was first produced during 1934-1937 in a three-volume edition, it had been intended to issue a complete revision every five or six years, but the recent war not only frustrated this intention by its long duration, but also so largely diverted the work of chemists into certain highly specialised channels, many of them new, that progress in these branches was greatly accelerated. This is seen particularly in the dramatic advances made in biochemistry, and also in the preparation of many compounds which assumed importance either directly or indirectly out of war conditions. In consequence, the new edition required complete revision and considerable enlargement, and, in order to avoid unwieldiness, it has been issued in four volumes. In its preparation, the literature has been searched up to the end of 1950, but during the printing period every opportunity has been taken to include more recent work, and some later references, even up to 1953, have been made possible. Over 2500 compounds have been added to the original collection, while amendments and additions have been made to existing entries following upon the appearance of fuller or more accurate data and information.

The new arrangement is— Volumes 1 *Abadole* to *Cytosine*, 2 *D.A.* to *Hystazarin*, 3 *Ibogaine* to *NW-Acid*, and 4 *Obaculactone* to *Zymosterol*. The dictionary is its own index, since the compounds are arranged in strictly alphabetical order, but since this *per se* is inadequate, owing to the fact that most organic compounds possess more than one name, while questions of orientation introduce further complications, some further rules are introduced to facilitate use and economise time of search. Trade and proprietary names are generally not used for the principal references, except where they come within the general rule as being in common use, or where, usually because of complexity of structure, no other names are feasible. The name selected for a parent compound is, with few exceptions, retained for all its derivatives; for example, alizarin having been chosen for 1:2-dihydroxyanthraquinone, all the derivatives of the latter will be found described as those of alizarin. A generous number of cross-references, sometimes as many as three or four, are given, by means of which the principal references can readily be found.

The full constitutional formula is given for each compound, except for the cases of many compounds of the carbohydrate, steroid, and polyterpene classes, where it is represented by simple skeletal

structures and configuration by heavy or dotted lines in conformity with modern practice. It is to be noted that no one system of orientation of ring compounds has been followed exclusively, and it is praiseworthy that no new departures have been made from those already in use.

The literature references relate primarily to the best method of preparation, but in some cases publications are cited which refer to earlier methods of preparation, or which contain useful summaries or bibliographies. Where the original literature reveals numerous instances of divergent and sometimes contradictory data for the same compound, palpable inconsistencies have not been recorded, but where a choice could not be safely made, the alternative data, e.g. m.p., b.p., etc., are given in parentheses.

A warning, however, must be given to the beginner in organic research. Despite all the care and years of effort that have been devoted to make the present dictionary as complete as possible, the search for the novelty of a newly prepared compound must never be confined to one lexicon or similar source. For most British chemists, *Heilbron and Bunbury* will obviously be the first compilation to consult, but failure to find the compound there is no final guarantee of its novelty, although it will enhance hope that it is new. The same advice applies even more strongly to the search for the best recorded method of preparation of any particular compound, because all the editors and contributors combined, although each is a distinguished chemist in his own field or fields of activity, cannot decide for all compounds in the vast domain covered by the dictionary. The reviewer also can spot mistakes or omissions only when they fall within his own experience, and accordingly he has checked up on some of the data against his own publications. He has been gratified to find agreement in a large number of the cases examined, but many omissions and out-of-date references have also been revealed, of which the following are only a small selection, with date of publication in parentheses: some compounds omitted are— 2:2'-diamino-5:5'-ditolyl di- and tri-sulphide (1912), 4:4'-diamino-diphenyl tri-sulphide (1925), 3-fluoro-4-nitrosophenol (1940), and *o*-fluoro-*p*-benzoquinone. All the data and references to the *m*-halogeno-*p*-nitrosophenols and *m*-halogeno-*p*-benzoquinone amines exhibit inadequate care in compilation, inaccuracy, and confusion (1922, 1923, 1925, 1929, 1932, *et seq.*). Reliable methods of preparation, unrecorded, concern the following compounds—*p*-nitrophenylhydrazine (1926), *m*-hydroxybenzaldehyde (1925, 1926), the best separation of 1:5- and 1:8-dinitronaphthalenes (1933), and 1:3-dinitronaphthalene (1943) (the method which made this potentially important compound available for the first time). Further, the patent reference given for the preparation of *m*-bromophenol (1922) should have been included under the more important *m*-chlorophenol and also under *m*-iodophenol, since it is not only a general method but

the only one which gives a good yield ( $> 90\%$ ). There are many other similar omissions of the reviewer's own work, and they serve to emphasise his earlier warning, since other chemists are bound to find similar defects in their own fields.

Nevertheless, this dictionary is a monumental work on which all concerned in its production are to be congratulated, and for which organic chemists in general and research workers in particular will be deeply grateful.

H. H. HODGSON

### **Lehrbuch der Organischen Chemie**

By Paul Karrer. 12th edition (revised and enlarged) 1954. Pp. xx + 949 + 6 plates. Stuttgart: Georg Thieme Verlag. Price, DM 59.70.

This textbook, the first edition of which appeared in 1927, was originally compiled from lectures delivered to students in the University of Zürich, and its success, which has been international in character, is testified by the fact that the 12th German edition is now to hand. Thorough revision has been necessary in view of an unprecedented expansion of the subject, and many of the former sections have had to be recast entirely. In particular, the treatment of reaction mechanisms from the standpoint of modern electronic theory has been adopted throughout and has required considerable space, part of which has been gained by suitable curtailment of other sections. Emphasis, however, is laid on experiment as the safest foundation for further progress in chemistry.

The new edition broadly resembles the original in the method of presentation, the important compounds being given their method of preparation with a proof of contribution. As one would expect from a successor and former pupil of the great Werner, stereochemical problems are treated with the thoroughness of the Master.

The book is divided into four main parts—(I) Aliphatic (21 chapters), (II) Carbocyclic (37 chapters) with further subdivision into (A) Aromatic and (B) Alicyclic, and (III) Heterocyclic Compounds (14 chapters). Part IV deals with Organic Compounds containing Isotopic Elements—heavy hydrogen, carbon, heavy oxygen and nitrogen. A subject index in three columns occupies 57 pages. The plan of the first edition has been generally adhered to, the groups of compounds being arranged accordingly to the nature and number of the functional groups, and the author states his conviction for its general utility. As a consequence, high polymers, for example, are not treated in a separate chapter but appear in their individual appropriate setting, e.g. the polysaccharides in I. The dyes mostly appear in IIA, but the phthalocyanines are in III. Obviously the various classes of dye receive only brief mention, most space being allotted to Indigo, but a few typical examples are included of the Indanthrens, as of the other main dyes.

It must be remembered that the book is primarily a text for the general student of organic chemistry, so that one can expect a subject like quinoneoxime-nitrosophenol tautomerism to be restricted to a brief mention, but to dismiss the nitronaphthyl-

amines as of no particular interest (p. 458), and to omit the general method published in 1933 by which *o*-phenylenediamine was tetrazotised (1935), 2:4-dinitro-1-naphthylamine first diazotised, and most other diazotisation problems solved, is doubtless due to the author's preoccupation with other important branches of the subject. This appears to be further reflected in his chronological list of outstanding discoveries since 1760, where 1856 is assigned to the first synthesis of methane by Berthelot, while Perkin's discovery of mauve is ignored, although it appears in somewhat perfunctory fashion on p. 597 of the text. Further, it is almost incredible that Willstätter's name appears only once in this list, and that for the pure preparation of the first anthocyanins, while the usual mistake is made of giving Böttiger the credit of discovering the first direct cotton dye, cited as Congo Red, in 1884, whereas it was preceded by Walter's discovery of Sun Yellow in 1883.

Nevertheless, this book is a monumental work, beautifully executed in arrangement, printing, and paper, although unfortunately for handling it weighs 5 lb. Few chemists will be able to resist purchasing it for their own use, and no teacher need hesitate before recommending it to his pupils. Many are the textbooks in organic chemistry, but this one is really great. An English translation will be welcome.

H. H. HODGSON

### **Organic Analysis**

#### **Volume 1**

Edited by J. Mitchell, I. M. Kolthoff, E. S. Proskauer, and A. Weissberger. Pp. viii + 473. New York and London: Interscience Publishers. 1953. Price, £8s. 0d.

The reader who feels that the world is already well filled with books on analysis, and that this is probably just another piece of coal on its way to Newcastle, could scarcely be more mistaken. The names comprising the Editorial Board and contributors are in themselves a hint of the excellence which the reader may confidently expect, and the whole publication bears the stamp of authority and of unity of purpose. It must be freely admitted that your reviewer has spent two months trying to find fault with this book, and has failed to arrive at any adverse criticism which might not fairly be termed carping.

The book, which the Editors announce as the first of a series, is in the form of monographs dealing with the analysis of individual organic groupings (if we may include active hydrogen amongst these), each being written by an acknowledged expert in his field—hydroxyl groups (Mehlenbacher); alkoxyl groups (Elek); the  $\alpha$ -epoxy group (Jungnickel, Peters, Polgar, and Weiss); organometallic compounds for the determination of active hydrogen (Wright); diazomethane for the determination of active hydrogen (Arndt); carbonyl compounds (Mitchell); acetals (Mitchell); organic sulphur groups (Dal Nogare); and spectroscopic functional group analysis in the petroleum industry (Coggeshall). The last-named section is the only one directed to a limited field of application. As a

whole the book may be considered as valuable to everyone from those at the ante-graduate stage to those to whom organic analysis is bread and butter; even to the expert analyst it constitutes a most useful review of available methods, in which modern developments in techniques such as those of spectroscopy, chromatography, and polarography take their due place. The bibliographies contain, collectively, over 1000 references to world literature.

The general method of contributors is to present a background in which the underlying theoretical principles are expounded fully, and to assess the relative importance, scope, and value of the available methods of determination; those of real significance are given in full experimental detail, an excellent feature of which is the explanation of the purpose of every operation and manipulation. Although there is clear evidence throughout of a firm editorial policy, the writers have properly allowed their presentation to be shaped by the nature of their material (a lesson which Science may profitably learn from Art); thus, the chapter on the use of diazomethane for the determination of active hydrogen is an orderly woven fabric of hitherto loose and scattered threads rather than a handbook of methods, whilst Elek's chapter on the determination of the alkoxy groups is primarily practical, and deals with every refinement of experimental procedure within a less diverse field.

The determinations of xanthate, hydroxyl, and alkoxy groups in cellulosic materials are specifically dealt with, and there are numerous references to and descriptions of methods of analysis as applied to carbohydrates, dye intermediates, and many other classes of substance of direct interest to the textile technologist. The analysis of epoxy resins, which is well covered, is particularly welcome in view of the currently growing interest in these materials in the fields of surface coating, and the stabilisation of chlorine-containing polymers such as P.V.C., which now find such wide application in fabric coating.

Perfection in a text-book is virtually a myth, and a brief citation of the observed shortcomings of this book is probably the best testimonial to its high quality. A few of the diagrams leave something to be desired, and would have been better had their size not been so reduced. The English is occasionally somewhat obscure; here are two examples—"Formic acid, however, is sufficiently strong so that a reaction between it and an alcohol proceeds to completion."; and "... fractional recrystallisation is in order since the solubilities of the esters show variations in melting points, thereby facilitating identification". The generally admirable detail is occasionally pushed a little too far, and it is mildly irksome to the adult chemist to be told that "A 5% aqueous solution of reagent quality cadmium sulphate is prepared by dissolving 5 g. of cadmium sulphate in 100 ml. of water." These small things apart, the overall standard of typography, lay-out, English, and make-up is very high; omission of the terminal "s" from "two-thirds" on page 80 is the sole error we have detected though a few others may have escaped us in the same way

in which they have escaped the authors, editors and proofreaders.

It is, in conclusion, perhaps amusing to speculate as to the exact stage in the journey to immortality at which an originator's name passes into such common currency that it loses its initial capital (as with "grignard" and "bunsen"), whilst such recognition is still denied to many other "household" names such as Wij, Schiff, and Zeisel. Fame is a fickle jade.

J. W. DUARTE

### Fluorescence Analysis in Ultra-violet Light

By J. A. Radley and Julius Grant. Fourth edition 1954. Pp. xvi + 560 + 72 Fig. London: Chapman & Hall Ltd. Price, 52s. 6d.

Fluorescence analysis is an indispensable addition to the many delicate chemical and physical techniques now available to the analytical chemist, and this book gives virtually a complete account of almost all its uses. It is divided into two parts, dealing respectively with theory and technique, and applications. The first describes the theoretical background and the methods of producing, measuring, and using ultraviolet radiation, and includes many illustrations, diagrams, and full descriptions of modern lamps and analytical apparatus.

The second section of the book, describing the multitudinous uses of fluorescence, is much the larger of the two and includes chapters on agriculture, bacteriology, botany, constructional materials, drugs, foods, fuels and lubricants, inorganic and organic chemistry, leather and tanning, legal and criminological work, medical and biological science, minerals and gems, museum work, the rubber industry, and water and sewage. Readers of this *Journal* will be more particularly interested in the chapters on paper and cellulose materials, paints and varnishes, dyes, and textiles, with their very detailed descriptions of the methods of identification of fibres, colouring matters, and finishing agents, and the detection of damage, faults, or stains in fabrics.

The book is well worth perusal, however, by any scientist whatever his interests may be. It will introduce him to many analytical methods and tests which are sure to stimulate ideas and may well suggest to him new lines of investigation, and it will also warn him of the many difficulties which lie in the way of the application of this technique, which appears at first sight so simple and elegant. The illustrations in themselves, taken from photographs of fluorescent specimens of many types, from fossils to "invisible ink", are an attractive feature.

The value of this work to every user of fluorescence analysis is sufficiently evidenced by the fact that it has run through three editions and two reprintings since it was first published in 1933. The enormous amount of information which has been sifted by the authors is shown by the extent of the references to the original literature; these number nearly 3500, covering the work of some 2800 authors. No-one, in fact, who uses fluorescence analysis in any shape or form can afford to be without a copy of this book on his shelves.

The volume is attractively printed on good paper.

C. H. GILES

## Abstracts from British and Foreign Journals and Patents

The titles of abstracts may be modified. Abbreviations of names of firms are listed in J.S.D.C., 68, 23 (Jan. 1952), and also, together with symbols and the periodicals abstracted, in the annual index

### I—PLANT; MACHINERY; BUILDINGS

#### PATENTS

#### Steaming Synthetic Fibres. Chemstrand Corpn.

BP 705,101

Apparatus is described for the continuous treatment of synthetic fibre by high pressure steam. W. G. C.

#### Package Dyeing. E. A. Stienen. USP 2,637,992

A spindle assembly comprises a solid rod threaded into a tapered socket having a solid end portion in which the rod is embedded and a longitudinal bore with transverse peripheral openings for dye liquor to pass through. The socket is threaded on its outer surface so that it can be screwed into the shell of a dye carrier and at its inner end has a seat for a perforated tube on which yarn is wound. The longitudinal bore of the socket is not less than the inner diameter of the perforated tube while the total cross-sectional area of the transverse peripheral openings is greater than that of the perforated tube, thereby greatly increasing the volume of liquor that may be introduced into the spindle.

USP 2,637,993

The density of the package and so passage of the liquor through it is controlled by placing compression plates at the ends of the perforated tube. The plates have a mount to fit on the end of the tube and an annular zone to engage the sides of the package, this zone being flared towards the other end of the package from the tube to the outer periphery of the package so as to apply progressively increasing compression at the side of the package from the inner to the outer peripheries thereof. C. O. C.

#### Impregnating Textiles. Dayton Rubber Co.

USP 2,637,892

Apparatus which gives extremely uniform impregnation has a slasher roll which is coated with a layer of spongelike structure composed of channels, each channel consisting of interconnected regularly shaped pores. These pores are the same shape as water-soluble particles compounded with the material of the layer from which they are removed by dissolution in water. C. O. C.

#### Tenter Frame Washing System for a Mercerising Range. Springs Cotton Mills. BP 706,085

The wash water is independently recirculated at a number of washing stations in parallel from a common wash water supply having a relatively low throughput. This gives more effective washing than has hitherto been possible, the wash water discharge containing 10% NaOH. C. O. C.

#### Tenter. Famatex.

BP 707,161

A combined clip and pin tenter chain link which is not dangerous either to the fabric or the operator and which can be used in a machine employing jets of drying air. C. O. C.

#### Curved Expander Rolls. J. D. Robertson. BP 706,230

A description of better-wearing rolls requiring less tensional force for their rotation. C. O. C.

#### Automatic Weft Straightener. Marshall Field & Co. USP 2,638,656

The fabric is marked at regular intervals with a material invisible under ordinary light but which becomes luminescent under radiation projected from a source placed above the fabric, the radiation so emitted by the material then actuating photoelectric means which control the action of electric motors which adjust the fabric so as to straighten the weft. C. O. C.

#### Drying and Conditioning Fabrics. Samcoo Holding Corpn. USP 2,637,991

A machine in which several pieces of tubular fabric can be treated simultaneously. C. O. C.

#### Screen Printing. Roto-Matic Screen Printers.

USP 2,637,269

The squeegee is slowed down before reversal or stopping by means of a variable speed transmission in its drive and by sharply increasing the speed reduction just before the squeegee reverses or stops. C. O. C.

#### Spray Varnishing of Hides and Skins. Conceria Alta Italia Giraud, Ammendola & Pepino. BP 706,156

An endless conveyor carries the hides beneath sprayers mounted together with a light projector-photoelectric cell which controls the sprays. C. O. C.

#### Pelleting Carbon Black. Phillips Petroleum Co.

USP 2,638,625

Apparatus for forming chain structure carbon blacks into pellets. C. O. C.

#### Pelleting Furnace Black. Columbian Carbon Co.

USP 2,639,225

Production of Metal Powders by Atomisation (IV p. 252).

### II—WATER AND EFFLUENTS

#### PATENTS

#### Water Treatment Proportioning Device. ICI.

BP 705,068

Water treatment chemicals are fed to a water reservoir in substantially the same proportion as the water fed by means of a constant level stock tank having an overflow and a displacement float. The float is supported by a bellows located at the bottom of the reservoir; contraction of the bellows due to increased weight of water in the reservoir displaces the float and causes an efflux of chemicals. J. W. B.

#### Apparatus Containing Ion-Exchange or other Beds of Granular Material. Permutit Co. BP 705,402

A system for collecting or distributing liquid in a tank containing granular material consists of a plate or series of pipes with openings for the passage of liquid from one face of the plate to the other, or to and from the pipe interiors, and strips of rigid material covering the openings and spaced away from their mouths so as to leave gaps through which liquid, but not granular material, can pass. J. W. B.

### III—CHEMICALS; AUXILIARY PRODUCTS; FINISHING MATERIALS

#### Reaction between Chlorous Acid and Glucose—Quantitative Stoichiometry and Evaluation of Reagent Decomposition. H. F. Launer and Y. Tomimatsu. Anal. Chem., 26, 382-386 (Feb. 1954).

Weakly acidic  $\text{NaClO}_2$  oxidises aldoses or aldehyde groups specifically and without side-reactions; the reaction with glucose as a model substance has been studied. In  $\text{H}_2\text{PO}_4^-$  phosphate buffers  $\text{NaClO}_2$  reacts with glucose in the ratio 3 : 1 without overoxidation with time, in accordance with the equation—



The reagent decomposes during the reaction, but this can be allowed for by assuming that it proceeds at a rate which is proportional to the geometric mean of the  $\text{ClO}_2^-$  concn., a simple expression being derived which is valid over the ranges 0.000004–0.0003 M. glucose, 0.0005–0.0032 M. chlorite, and pH 2.4–3.4 at 50°C. Glucose may be determined over the range 0.6–50 µg. per litre under the conditions described. It is shown how the effects due to the sensitivity of the reagent decomp. to light and to ionic strength may be controlled. All experimental details are given. J. W. D.

#### Carboxymethyl Cellulose—its Textile Uses. O. Wurz and E. Wurz. Textil-Rund., 8, 557-560 (Oct. 1953).

A review. The addition of 0.1% (on wt. of soln.) benzoic acid stabilises against bacteria. When used with sulphur dyes, skin formation was traced to small amounts of Fe or Mn, which can be prevented by addition of, e.g. Aquamollin (CFM). Carboxymethyl cellulose cannot be used in conjunction with the salts of certain metals, such as Cr, Zn, Sn, Al, and Pb, which also produce insol. cpd. H. E. N.

## PATENTS

**Surface-active Compositions.** Anglo-Iranian Oil Co. and Distillers Co. BP 706,604

A detergent composition particularly useful for washing cotton and scouring wool comprises a mixture of a soap and/or synthetic organic detergent with cyclohexyl carbinol or a substitution derivative having one or more alkyl groups attached to the naphthonic ring. The weight ratio of the soap-detergent to the carbinol must lie substantially between 0.5:1 and 2:1. J. W. B.

**Synthetic Detergents.** Lankro Chemicals. BP 706,570

*N*-β-Hydroxyethylamides of saturated or unsaturated fatty acids of 8-18C are converted into the corresponding sulphuric acid half-esters by treating them with H<sub>2</sub>SO<sub>4</sub> (>100% conc.) in presence of a C<sub>6</sub>-C<sub>12</sub> alkylaryl monosulphonic acid as solvent for the half-esters. The products have good detergent and surface-active properties. C. O. C.

**Synthetic Detergents.** Monsanto. BP 705,658

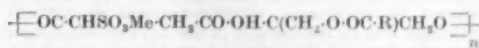
A detergent composition comprises an oily liquid to semi-liquid ethylene oxide condensation product of a compound containing an active hydrogen atom and a hydrated alkali metal salt, the hydration having been effected in presence of the condensation product and the weight ratio, prior to hydration, of salt to condensation product being from 1:1 to 10:1. The condensation product may be ethylene oxide with a mercaptan having at least 6 C atoms, or with tall oil. The salt may be hydrated tetrasodiumpyrophosphate or hydrated sodium carbonate. The oily condensation product is well retained by the solids, leading to good powdering and flowing properties. J. W. B.

**Textile Lubricants.** Standard Oil Development Co. BP 705,056

A lubricant suitable for both wool and textile machinery, with excellent scourability, comprises a mineral oil containing a minor proportion of two non-ionic type detergents, one being polyoxyalkylene derivatives of alkylated phenols and the other being polyoxyalkylene derivatives of aliphatic acids. A stabiliser such as a sodium mahogany sulphonate may also be added. J. W. B.

**Softening Agents.** American Cyanamid Co. USP 2,637,663

Compounds of formula



(R = aliphatic radical of 11-17C; Me = salt-forming cation; n = 2-10) are excellent softening agents which are unaffected by heat. C. O. C.

**Quaternary Ammonium Salt Derivatives of Alkylalkoxysilicon Compounds as Water-repellent Agents.** Deering Milliken Research Trust. USP 2,637,623

Textiles of all types are rendered water repellent by treatment with a solution or dispersion of a compound of formula R<sub>4</sub>Si(-O-Y-N(tert.)X)<sub>2-20</sub> (R = same or different, satd. or unsatd., subst. or unsubst., aliphatic, aromatic or cycloaliphatic or an -(O-SiR<sub>3</sub>)<sub>y</sub>-(O-Y-N(tert.)X)<sub>2-20</sub> linkage (y = 1-20); n = 1-3; N(tert.) = residue of a tertiary amine; X = an anion) and then treated so as to cause chemical reaction between the compound and itself and/or the fibres, e.g. acid or alkali treatment and/or heating or baking. The treatment also improves the softness, dimensional stability, flame-resistance, and crease-resistance. C. O. C.

**Flame- and Glow-resisting Agent.** Monsanto. USP 2,637,704

Heating a mixture of a polymeric phosphonitrilic chloride, ((PNCl<sub>2</sub>)<sub>3</sub>)<sub>n</sub>, chloroaniline and an ammonio carbonic acid or ammonolysed carbonic acid or an organic carbonol, e.g. urea or guanidine carbonate produces a condensate which is insoluble in cold water and in aromatic hydrocarbons and which has pronounced flame- and glow-resisting properties. C. O. C.

**Rotproofing Composition.** Monsanto. USP 2,637,661

An aqueous solution containing copper-, cadmium- or zinc-ammonium ions, pentachlorophenate ions, non-ionised water-soluble pentachlorophenate, an alkylated methylol derivative of melamine or urea, and a water-miscible organic solvent for copper, cadmium and zinc pentachloro phenates, is used for permanently rot-proofing textiles. C. O. C.

**Aryl Mercuric Rotproofing Agent.** Standard Oil Co. USP 2,637,677

Compounds of formula



(Z = benzene or naphthalene; X = acid radical; R = H or unsat. aliphatic group of 1-18C; A = a polyoxyethylene ether, polyoxypropylene ether or polyoxyethylene-propylene ether chain containing 4-25 oxyalkylene groups) are water-soluble liquids having excellent rotproofing properties and suitable for application to textiles, leather, paper, etc. C. O. C.

**Tanning Agents from Alkali-Lignin.** Champion Paper & Fibre Co. USP 2,639,988

A highly reactive tanning agent is formed when aqueous-chlorine-treated alkali-lignin (Cl content 15-30%) is mixed with an alkali-metal sulphite, bisulphite or metabisulphite and at some stage treated with water. C. O. C.

**Shellac Substitute.** Standard Dyewood Co. BP 703,407

Catechol tannins, e.g. Quebracho extract, are reacted under alkaline conditions first with chloroacetic acid (1-2 mol. per mol. tannin) and finally with benzyl chloride (< 1 mol. per mol. tannin) and the product is converted to the free acid by acidification. BP 704,408

The process of BP 703,407 (above) is modified to include, as an intermediate stage, condensation with formaldehyde under alkaline conditions.

Surface coating resins are prepared by dissolving the acid resinous condensation products of BP 703,407-8 (above) and/or a protein, and/or *N*-ethyl toluenesulphonamide, and/or carnauba wax in a solvent, e.g. methanol and toluene or an aqueous solution of a volatile base such as ammonia. E. C.

**Backsizing composition for Carpets.** Alexander Smith. USP 2,637,095

A backsizing composition based on an elastomeric resin, preferably a styrene-copolymer latex, gives the fabric backing of the carpet the same properties as are obtained by use of special backing yarns and weaves. It improves the dimensional stability, retention of handle of the carpet on being shampooed and resistance to fraying of cut edges. C. O. C.

**Alkali-Lignin Tanning Agent.** Champion Paper & Fibre Co. USP 2,640,052

The "black liquor" obtained from alkaline digestion of wood, etc. is first acidified to precipitate the sodium lignate. This is dissolved or suspended in water and treated with chlorine with vigorous agitation until a product containing 15-30% chlorine is obtained. This product is then treated with an alkali, this treatment resulting in part of the chlorine content being replaced by OH to yield a tanning agent which is soluble at pHs down to below those of vegetable tanning liquors and which contains 40-70% tannin based on the American Leather Chemists Association's hide powder test. C. O. C.

**2:2' - Dihydroxy - 4:4' - dialkylbenzophenones—Absorbents for Ultraviolet Radiation.** General Aniline. BP 706,151

2:2'-Dihydroxy-4:4'-dialkylbenzophenones have excellent ultraviolet radiation absorbing properties, their efficiency at 400 mμ being about 12 times that of any other compound now in use for that purpose. They are compatible with numerous film-forming plastics, resins, gums, waxes, etc. C. O. C.

## IV—RAW MATERIALS; INTERMEDIATES; COLOURING MATTERS

### Oxidation of Some Aromatic Amines with Persulphate. E. Boyland and P. Sims. *J.C.S.*, 980-985 (March 1954).

The action of alkaline persulphate on a number of substituted aromatic amines is reported, when in all cases *o*-aminophenyl sulphates are formed irrespective of the orienting influence of other functional groups. Under the same experimental conditions, persulphate does not yield sulphuric esters with benzoic acid, nitrobenzene, toluene, benzene, naphthalene, pyridine, or cyclohexylamine. Whereas aniline, 2-naphthylamine, and dimethylaniline readily form sulphuric esters, no effect occurs with acetanilide and 2-acetamidonaphthalene. The reaction differs from the Elbs persulphate oxidation of phenols, where substitution occurs mainly in the *para* position, and less readily in the *ortho* position when the *para* position is blocked.

H. H. H.

### ortho-Hydroxylation of Phenols. IV—Pyrogallols J. D. Loudon and L. A. Summers. *J.C.S.*, 1134-1137 (March 1954).

Pyrogallol and its 4- and 5-methyl and 4:5- and 4:6-dimethyl deriv. are prepared from phenol and its appropriate homologues, via 2-aryloxy-3:5-dinitrobenzenes and the derived 2-2'-hydroxyaryloxy- and 2-(2':6'-dihydroxyaryloxy)-3:5-dinitrobenzenes.

H. H. H.

### Azoic Dyes and their Intermediates. IV—Action of Nitrous Acid in the Nitration of *p*-Methylanisole. S. Yura, A. Nagasaka and I. Tanaka. *J. Chem. Soc., Japan, Ind. Chem. Sect.* 55, 436-8 (1952); V—Preparation of Red Bases which give Stable Neutral Diazo Solutions. S. Yura and A. Inoue. *Ibid.* 439-440; *Chem. Abs.*, 48, 3031 (10 March 1954).

IV—The nitration of *p*-methylanisole in dil.  $\text{HNO}_3$  is catalysed by  $\text{HNO}_2$ , nitroso-*p*-methylanisole being formed as an intermediate in the reaction. The principal product of the nitration of *p*-methylanisole with 98%  $\text{HNO}_3$  (containing  $\text{HNO}_2$ ) in glacial acetic acid at 15°C. is 2-nitro-4-methylanisole.

V—*p*-Aminobenzanilide and its chloro and methyl-derivatives (I), *p*-amino benzene sulphonanilide and its chloro and methyl derivatives (II), *N*-cyclohexyl-*p*-aminobenzamide (III), and *N*-cyclohexyl-*p*-aminobenzene-sulphonamide (IV) were prepared. Their neutral diazo solutions are much more stable than that of Fast Scarlet G Base, i.e.  $\text{IV} > \text{III} > \text{II} > \text{I} > \text{Fast Scarlet G Base}$ . Among I and II the influence of the substituted group on stability is  $\text{Cl} > \text{H} > \text{CH}_3$ . The base of IV gives brilliant red with Naphthol AS and has good fastness to light.

C. O. C.

### Some $\beta$ -Furylstyrene Dyes. A. Mangini and G. Modena, *Ricerca sci.* 23, 1649-1651 (1953); *Chem. Abs.*, 48, 3031 (10 March 1954).

$\beta$ -(2-Furyl)-2:4-dinitrostyrene with Baumgarten's reagent (pyridinesulphonic acid) and  $\text{Na}_2\text{CO}_3$  gives the Na salt of  $\beta$ -(5-sulpho-2-furyl)-2:4-dinitrostyrene. This on reduction with glucose, phenylamine or other aromatic bases gives yellow-brown dyes which are not fast to washing. The reduction product, diazotised on the fibre and coupled with phenol, amine, 3-hydroxynaphthoic acid or Schaffer's acid, gives fast reds.

C. O. C.

### Characteristic Infrared Absorption of the Triphenylmethyl Group. S. Pinchas and D. Samuel. *J.C.S.*, 863-864 (March 1954).

The spectra of 13 compounds which contain the triphenylmethyl group all show a medium-strength band at  $\sim 1185 \text{ cm}^{-1}$  and a weak band at  $\sim 1280 \text{ cm}^{-1}$ , which seem to be characteristic of this group. These bands are assigned to the stretching modes of vibration of the  $\text{C}-\text{C}_6\text{H}_5$  bonds.

H. H. H.

### Triphenylmethane Derivatives from Condensations of Phenols with Hydroxybenzaldehydes. J. E. Driver and J. B. Sousa. *J.C.S.*, 985-989 (March 1954).

Previously, only resinous products have been obtained by the condensation of phenol with *o*- or *p*-hydroxybenzaldehyde, but it is now shown that, at relatively low temp. and in a suitable medium, e.g. glacial acetic acid,

triphenylmethane deriv. are readily prepared by the acid-catalysed condensation of phenol and the cresols with various aromatic hydroxyaldehydes, although in most of the reactions amorphous resins remain the chief product. The mechanism of reactions of this type is considered, and evidence is given of the intermediate formation of a dihydroxydiarylmethanol in two condensations, and of the reversibility of some of the changes involved.

H. H. H.

### Action of Bromine and Oxidising Agents on Pyrrole Blues. G. Castorina. *Il Farmaco, Ed. sci.* (Pavia), 8, 658-683 (1953); *Chem. Abs.*, 48, 3031 (10 March 1954).

Oxidation of simple pyrrole blue with  $\text{Br}_2$ ,  $\text{K}_2\text{S}_2\text{O}_8$ ,  $\text{FeCl}_3$ , benzoyl peroxide,  $\text{C}_2\text{H}_5\text{NO}_2$  and  $\text{NH}_4\text{NO}_2$  (to supply 1 atom O per mol. of dye) yields a stable soln. of a reddish violet oxidation product. Neither evaporation nor precipitation enabled a crystalline compound to be isolated. Reducing agents ( $\text{Na}_2\text{S}_2\text{O}_4$ , Zn dust) reformed the original blue dye from an acetic acid soln. of the above oxidation product, more energetic reduction forming the leuco compound.  $\text{H}_2\text{S}$  in acetone leads only to reduction to blue.  $\text{Na}_2\text{S}_2\text{O}_8$ , glucose and ascorbic acid lead to the leuco base.  $\text{H}_2\text{S}$  is most suitable to prepare the blue dye from the dehydro derivative. The purified product is amorphous and more soluble in acetic acid and pyridine than the original compound. Titration of the dye dissolved in acetic acid with an acetic soln. of Br shows change to red-violet when two atoms of Br have been added. Further addition of Br produces change to yellowish orange.

C. O. C.

### Recent Progress in Sensitising Dyes. S. Kimura. *Sci. Pubs. Fuji Photo Film Co., No. 1*, 76-94 (1953); *Chem. Abs.*, 48, 2497 (10 March 1954).

A review of the constitutions, preparation and application of sensitising dyes. 182 references.

C. O. C.

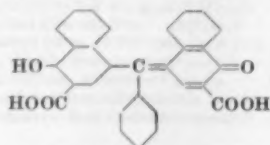
### Relationship between the Absorption Spectra and the Chemical Constitution of Dyes. XXVII. *cis-trans*-Isomerism and Hydrogen Bonding in Indigo Dyes. W. R. Brode, E. G. Pearson and G. M. Wyman. *J. Amer. Chem. Soc.*, 76, 1034-6 (20 Feb. 1954).

Examination of the ultraviolet and visible absorption spectra of indigo and six halogen-substituted indigo dyes showed that they differ from thioindigo dyes in that there is no photochemical isomerisation. This can be ascribed to there being preferential stabilisation of the *trans* configuration by hydrogen bonding. This hypothesis is confirmed by occurrence of phototropism in *NN'*-diacetylindigo and by comparison of the infrared absorption spectra of indigo and thioindigo. Introduction of halogen atoms has the usual bathochromic effect accompanied by increase in intensity of absorption, bromine causes greater shift than does chlorine and thus effect of halogens appears to be cumulative. 4:4'-Dichloroindigo has unusually intense absorption and this may be due to the position rather than the nature of the substituents, although the reasons for this are not clear.

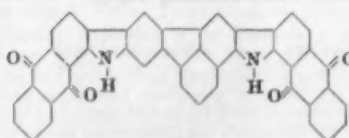
C. O. C.

### The Swiss Contribution to Colour Chemistry. R. Wizinger. *Textil-Rund.*, 8, 460-472 (Sept. 1953).

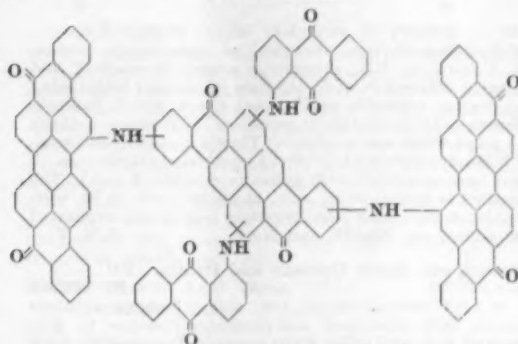
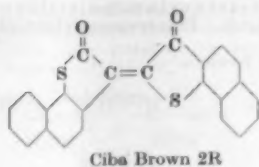
A review, including the following less generally available formulae—



Naphthochrome Green G



Cibanone Red Brown 2BR



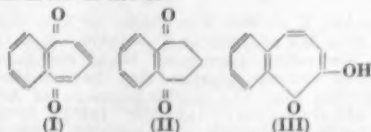
The formulæ given for Polar Blue G, Ciba Violet 3B, and Ciba Red G differ slightly from those recorded in Venkataraman's book. H. E. N.

**5-Aminoindole.** J. Harley-Mason and A. H. Jackson. *J.C.S.*, 1158-1159 (March 1954).

Oxidation of 2-(5-amino-2-hydroxyphenyl)ethylamine by silver oxide gives 5-aminoindole. H. H. H.

**Tropolones. VIII—Synthesis and Properties of 7-Hydroxybenzocycloheptatriene-3-one.** G. L. Buchanan. *J.C.S.*, 1060-1063 (March 1954).

The bicyclic compound (I), named in the title, is synthesised from benzocycloheptene-3:7-dione (II), and its properties are compared with those of  $\alpha\beta$ -benzotropolone (III), which it resembles in that it gives a 3:5-dinitrobenzoate, an oily acetate, a yellow picrate, and a red-brown colour with  $\text{FeCl}_3$ , couples with diazotised aniline to produce a crimson dye, and forms unstable salts with  $\text{HCl}$  and  $\text{HClO}_4$ ; it differs in that it forms a 2:4-dinitrophenylhydrazones, is almost insol. in non-polar solvents, fails to form a copper complex, and is not affected by fusion with  $\text{KOH}$  at  $220^\circ\text{C}$ .



H. H. H.

**Aromatic Hydrocarbons. LXVI—Diperinaphthylene-anthracenes.** E. Clar, W. Kelly, and J. W. Wright. *J.C.S.*, 1108-1111 (March 1954).

1:9-5:10- and 1:9-4:10-Diperinaphthyleneanthracene are prepared by an unambiguous method from 1:5- and 1:4-dichloroanthraquinone respectively, and show similar properties. Their absorption spectra are compared with quantum-mechanical predictions by Dewar. H. H. H.

**Advances in Predicting the Spectra of Condensed-ring Systems.** J. R. Platt. Pittsburgh Conference on Analytical Chemistry and Applied Spectroscopy. *Anal. Chem.*, 26, 433 (Feb. 1954).

Relations among the allowed and forbidden transitions and among the singlets and triplets in the excited electronic states of benzene, naphthalene, and other aromatic mol. are similar to those found for the energy states of rare gas atoms. Several new molecular-orbital and free-electron theoretical methods permit simple but fairly accurate predictions of the wavelengths and intensities of absorption for these mol. The wavelength shifts between two isomeric ring systems like anthracene and phenanthrene can be computed, and a start has been made on the problem of predicting wavelength shifts produced by

chemical substitution of the mol. Intensity changes in certain bands with chemical substitution follow a simple vector rule, from which it is possible to obtain experimental information about the symmetry of the wave functions of the excited states. J. W. D.

**Polycyclic Systems. VI—8-meso- $\alpha$ -Naphthanthr-10-one and 2:3-8:9-Dibenzoperylene.** E. A. Braude, J. S. Fawcett, and A. A. Webb. *J.C.S.*, 1049-1059 (March 1954).

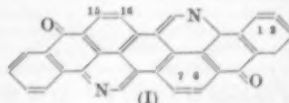
A novel route, based on the cyclisation of Diels-Alder adducts of substituted butadienes with various quinones to give condensed polycyclic benzenoid systems, is illustrated by unambiguous syntheses of meso- $\alpha$ -naphthanthr-10-one and 2:3-8:9-dibenzoperylene via 1-o-tolyl- and 1:5-di-o-tolyl-anthraquinone. The light-absorption properties of these and related compounds are recorded and discussed. H. H. H.

**Preparation of 2-Substituted Fluoranthenes.** N. Campbell and J. F. K. Wilshire. *J.C.S.*, 867-869 (March 1954).

A synthesis of 2-nitrofluoranthene from 1:2:3:4-tetrahydrofluoranthene (readily obtained by the reduction of fluoranthene) is described, as an example of a general method for the preparation of 2-substituted fluoranthenes. H. H. H.

**Synthesis of 9:18-Diazaisoviolanthrone.** J. King and G. R. Ramage. *J.C.S.*, 936-938 (March 1954).

The synthesis of 9:18-diazaisoviolanthrone (I) is described, and its structure confirmed by an alternative synthesis via 3-bromo-1-azamerobenzanthrone. The alkaline fusion of 1-azamerobenzanthrone gives I and not the expected 15:18-diazavioanthrone.



H. H. H.

**Vat Dyes of the Acenaphthene Series. III—Syntheses of Naphthostyryl and Anthanthrone from Naphthalimide.** T. Maki, S. Hashimoto and K. Kamada. *J. Chem. Soc. Japan, Ind. Chem. Sect.*, 55, 483-5 (1952); *Chem. Abs.*, 48, 3029 (10 March 1954).

Naphthostyryl was obtained in 56-5% yield by the Hofmann reaction between 1 mol. naphthalimide and 3 mol.  $\text{NaOCl}$ . 1:1'-Binaphthyl-8:8'-dicarboxylic acid was prepared by the diazo reaction of the product obtained by the hydrolysis of naphthostyryl. A 68% yield was attained in the diazo reaction by using cuprous ammonia soln. (prepared from  $\text{CuSO}_4$  and freshly precipitated metallic Cu (cuprous ammonia:Cu::2:1)). Anthanthrone was obtained by treating naphthostyryl with conc.  $\text{H}_2\text{SO}_4$ . C. O. C.

**Bixa Orellana: Annatto.** W. Diemair and D. Heuser. *Z. Lebensm.-Untersuch. u. Forsch.*, 97, 289-296 (1953); *Chem. Abs.*, 48, 2838 (10 March 1954).

Review with 31 references on geographical source, morphology, histology of the seeds of the annatto tree, culture, harvest, chronological history, and chemistry of bixin. C. O. C.

**Natural Dyewoods and their Application—Logwood.** H. R. Tisdale. *Amer. Dyestuff Rep.*, 43, 157-159, 164 (15 March 1954).

A brief review of the history, processing and chemistry of logwood followed by an account of its textile and non-textile applications. C. O. C.

**Iron Content of Melanin Granules Isolated from Pigmented Mammalian Hairs.** G. Laxer and C. S. Whewell. *Chem. and Ind.*, 127 (30 Jan. 1954).

Determination of Fe associated with melanin in brown and black hairs and wools is difficult, since the conc. acid used to separate the melanin granules from the protein matrix disrupts the melanin granules and probably removes Fe from the melanin-metal complex. A method is detailed in which a soln. containing phenol and thioglycolic acid is the extractant, which obviates the above difficulty;

the extracted melanin granules are morphologically undamaged. Iron contents of 0.149–0.538% are recorded for two hairs and three wools. Natural melanins isolated by this method absorb Fe from slightly acidic soln. of  $\text{FeSO}_4$ , suggesting that the Fe is associated in a non-ionic form. Since pigmented wools darken when treated with soln. of  $\text{Fe}^{2+}$  salts, differences in colour of fibres containing equal amounts of melanin may be partly due to variation in associated Fe content.

J. W. D.

**Structure of Natural Melanins.** J. L. Stoves. *Chem. and Ind.*, 158 (6 Feb. 1954).

A review and discussion of published work in this field. 11 references. J. W. D.

**A Glycosidic Dye and Several Colourless Constituents of the Japanese Wongsy *Gardenia grandiflora*.** K. Hayashi. *Proc. Imp. Acad. (Tokyo)*, 20, 311–7 (1944); *Chem. Abs.*, 48, 2838 (10 March 1954).

The yellow colouring matter present in Wongsy was identified as crocin. 1225 g. of incompletely dried seeds yielded 1 g. of pigment. C. O. C.

**Synthetic Iron Oxide Pigments.** P. J. Cautermanff. *Canadian Chemical Processing*, 38, 50–54 (Jan. 1954).

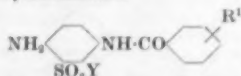
A brief history of the development and manufacture of synthetic iron oxide pigments is followed by an account of the manufacture, in Canada, of iron oxides by the Pennimann & Zoph method (*USP* 1,327,061 and 1,368,748) and the properties of the ferrite pigments. C. O. C.

## PATENTS

**Orange and Red Monoazo Acid Dyes.** Ciba.

BP 705,959

Sulphones and sulphonamides of monoacylated derivatives of *p*-phenylenediamine



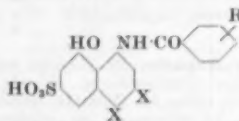
( $\text{R}^1$  = Alk or cycloalk of 3–8 C; Y = Ar or  $\text{N-R}^2\text{R}^3$  where  $\text{R}^2$  = Alk, cycloalk or Ar and  $\text{R}^3$  = H or Alk) are diazotised and coupled with a 2-naphthylamine-6-sulphonic acid (preferably  $\gamma$ -acid) to yield orange and red monoazo acid dyes. Thus 2:5-diamino-4'-methyldiphenyl sulphone is acylated with 1 mol. of *p*-*tert*-butylbenzoyl chloride, and the product diazotised with nitrosylsulphuric acid. After pouring on to ice, the diazo compound is filtered off and mixed with a finely-divided paste of  $\gamma$  acid. Coupling is effected by addition of sodium acetate and soda ash, to yield the azo dye which dyes wool bordeaux from an acetic acid bath.

E. S.

**Red, Monoazo Acid Dyes.** Ciba.

BP 705,961

*N*-acyl derivatives of 1:8-aminonaphtholdisulphonic acids



(R = Alk or cycloalk of 3–8 C; one X = H, the other X =  $\text{SO}_2\text{H}$ ) are alkali-coupled with a diazo compound of an amine free of *o*-OH groups or water-solubilizing groups to give red acid dyes. Thus 2-amino-4:2'-dichlorodiphenyl ether is diazotised and coupled with *N*-*p*-*tert*-butylbenzoyl-H acid in presence of soda ash. The monoazo compound so formed dyes wool bright bluish red from an acetic acid bath.

E. S.

**Yellow, Metal-complex Monoazo Dyes for Anodised Aluminium.** Ciba.

BP 703,949

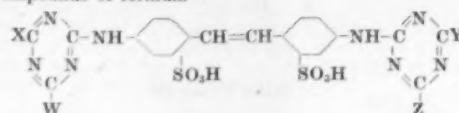
The cobalt and copper complexes of the monoazo compounds 2-aminophenol-4:6-disulphonic acid  $\rightarrow$  a 3-methyl-1-phenyl-5-pyrazolone are yellow dyes for anodised aluminium. The 1-phenyl group of the pyrazolone is free of sulpho groups and preferably contains 1, 2 or 3 Cl atoms. Thus the monoazo compound 2-aminophenol-4:6-disulphonic acid  $\rightarrow$  3-methyl-1-*o*-chlorophenyl-5-pyrazolone is heated at 70–75°C. The product dyes anodised aluminium golden yellow.

E. S.

**4:4'-Bis-(1:3:5-triazylamino)stilbene-2:2'-disulphonic Acids—Fluorescent Brightening Agents.** Ciba.

BP 705,406

Compounds of formula—



(W = primary or secondary alkyl- or alkylol-amino-,  $\text{N,N}'$ -dialkyl-alkylene diamino- or heterocyclic amino; X, Y and Z =  $-\text{NH}_2$ , primary or secondary amino, Y and Z being different from W, etc.) are fluorescent brightening agents for vegetable and animal fibres, which fluoresce blue to violet in ultraviolet radiation. Alternative methods of preparation are available. Thus 4:4'-diaminostilbene-2:2'-disulphonic acid (1 mol.) and monoethylamine (2 mol.) are condensed with cyanuric chloride (2 mol.). The product is heated in aq. soln. at 80–90°C. for 10 hr. with *o*-chloroaniline and the resulting compound separated by adding aq.  $\text{Na}_2\text{CO}_3$  and NaCl.

R. K. F.

**Brown, etc. Azoic Dyeings and Prints.** FH.

BP 706,586

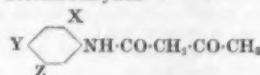
*N*-Acyl derivatives of 1:6- and 2:7-aminonaphthols couple with diazotised 4-aminodiphenylamines to give browns, and with other diazo compounds commonly used in azoic dyeing and printing to give golden-yellows, oranges reds and blues, and so may be used as coupling components in the production of white and coloured resists under a brown ground shade on vegetable fibres. Thus a cotton fabric is impregnated with a soln. of terephthaloyl- $\text{N,N}'$ -bis-1:6-aminonaphthol in aq. NaOH. After drying it is printed with a thickened paste containing aluminium sulphate and the  $\text{ZnCl}_2$  double salt of 3:5-bistrifluoromethylbenzenediazonium chloride, again dried, impregnated with a soln. of diazotised 4-amino-4'-methoxydiphenylamine and treated in dil.  $\text{H}_2\text{SO}_4$  at 60–70°C., so producing a golden-yellow resist on a brown ground.

E. S.

**Yellow Monoazo Pigments.** FH.

BP 705,590

Yellow monoazo compounds made by coupling a diazotised 2:5-dimethoxyaniline-4-sulphonarylamide (the aryl group = phenyl which may contain Alk, O Alk or Hal) with an acetoacetylride—



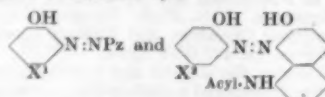
(X = Alk; Y = Hal; Z = OAlk; or X = OAlk; Y = Hal; Z = Alk or OAlk; or X = OAlk, Y = OAlk, Z = Hal), are yellow pigments. When incorporated into polyvinyl chloride plastics they have better fastness to bleeding than the similar pigments of BP 462,601 and 462,674 (*J.S.D.C.*, 53, 300 (1937)), in which the same acetoacetylrides are coupled with diazotised halogenoarylamines, e.g. chloroanilines. Thus 2:5-dimethoxyaniline-4-sulphonanilide is diazotised and coupled with an aq. suspension of acetoacet-4-chloro-2:5-dimethoxyanilide made by precipitating it from soln. in aq. NaOH with acetic acid.

E. S.

**Metal(Chromium)-complex Azo Dyes for Wool, Nylon, etc.** Gy.

BP 706,370

The chromium complexes from 1 atom of Cr and 1 mol. of each of two monoazo dyes—



(one X =  $\text{SO}_2\text{Alk}$  or  $\text{SO}_2\text{NR}^1\text{R}^2$ ; the other X =  $\text{NO}_2$ ,  $\text{SO}_2\text{Alk}$  or  $\text{SO}_2\text{NR}^1\text{R}^2$ ;  $\text{R}^1$  and  $\text{R}^2$  = H, Alk or hydroxyalkyl; Pz = the residue of a 1-aryl-3-methyl-5-pyrazolone coupled in the 4-position; other groups, but not water-solubilizing groups, may be present), dye wool, nylon, etc. brown from neutral or weakly acid baths. Thus 1 mol. of each of the monoazo compounds 2-aminophenol-4-methylsulphone  $\rightarrow$  1-*m*-chlorophenyl-3-methyl-5-pyrazo-



Dyes of formula  $R^1-CO-CCN:CH(CH:CH)_n-D-NR^2R^3$  ( $D = a$   $p$ -arylene group of the benzene series;  $R^1$  and  $R^2 = Alk$ ;  $R^3 = a$  sub. or unsub. benzene nucleus;  $n = 0$  or  $1$ ) are readily bleached by aqueous sulphites. C. O. C.

**Resin Dyes of the Cyanine Type.** Eastman Kodak Co. USP 2,639,282

A cyanine dye containing a hydroxyalkyl attached to one of the auxochromophoric N atoms is reacted with a vinylene polymer containing carboxylic halide and/or anhydride groups. The products are non-diffusing from gelatin. C. O. C.

**Readily Dispersed Sulphur Dye Preparations.** CFM. BP 705,936

Sulphur dyes can be obtained in dry, extremely fine form by treating them in presence of a protective colloid and a dispersing agent with only 10–20% of the weight of reducing agent that is normally necessary to obtain complete reduction of the dye. The products are non-dusting, have good fastness to stoving and when pasted with water yield a finely dispersed suspension. C. O. C.

**Solubilised Hydron Blue and Indo-Carbon Black Preparations.** Southern Dyestuff Corp. USP 2,627,450

Hydron Blue and Indo-Carbon Black in the form of filter press cake are dissolved in aq. alkali metal hydrosulphite with or without polysulphide, to give a liquid preparation containing the dye in a reduced ready-to-dye state suitable for package dyeing. Thus all parts being by weight, alkaline Hydron Blue press cake (100) is heated at 90°C. for 30 min. with 45% sodium hydrosulphide (84) in water (816). R. K. F.

**Isolating the Colouring Compounds from Caramel.** Union Starch & Refining Co. USP 2,637,655

The colouring compounds are precipitated from caramelised sugar by treating it with aqueous mixtures of alcohols, one of the alcohols being highly soluble in water and another regulating the solubility, e.g. a mixture of monohydric alcohols of 1–2 C and 3–5 C respectively. C. O. C.

**Lamellar Pigments of Ductile Materials.** Treffleries et Laminiers du Havre. BP 706,251

The pigment, e.g. metal or thermoplastic resin, is formed into a slurry which is then caused to flow repeatedly between surfaces in rolling and non-rubbing movement and which are resiliently pressed together. The products have high leafing and floating power. C. O. C.

**Production of Metal Powders by Atomisation.** H. A. Golwynne. USP 2,638,626–7  
USP 2,638,630

**Improving the Colour and Texture of Titanium Dioxide.** DuP. BP 702,807

Dry grinding  $TiO_2$  in presence of 0.5–5.0% of its weight of an aerogel, e.g. a silica aerogel, yields a product of extremely uniform texture and improved brightness, resistance to yellowing and durability in paint films. C. O. C.

## V—PAINTS; ENAMELS; INKS

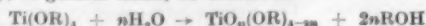
**Observations on the Optical Properties of Pigmented Films.** R. H. Kienle and C. Marosh. *J. Oil and Col. Chem. Assoc.*, 36, 619–645 (Nov. 1953).

Electron, and some optical, micrographs (33 in all) illustrate the influence on the optical properties of films, of the following pigment properties—refractive index, light absorption, particle shape and size, degree and method of deflocculation, weathering, gloss and surface imperfections. J. W. D.

**Esters of Titanium and their Use in Paint. I—Preparation of Polymeric Butyl Titanates. II—Hydrolysis and Film Formation. III—Formulation and Performance of Butyl Titanate Paints.** G. Winter. *J. Oil and Col. Chem. Assoc.*, 36, 689–694, 694–701, 701–708 (Dec. 1953).

I—Polymeric butyl esters of Ti have been prepared by partial hydrolysis of the monomer using various amounts

of water dissolved in butanol. The hydrolysis reaction may, in general terms, be written as—



Hydrolysis is quant. on addition of up to ~1.5 mol-equivalents of water; on further additions only a portion of the water added enters the reaction, a portion being absorbed and remaining in the solvent. The direct preparation of polymeric butyl titanate from  $TiCl_4$ , butanol, and water on a pilot-plant scale is described; yields of 80% have been obtained.

II—The rates of hydrolysis of monomeric  $n$ -butyl,  $sec$ -butyl,  $tert$ -butyl,  $n$ -hexyl,  $n$ -octyl, "alphanol", and "nonanol" Ti esters as well as those of a number of polymeric  $n$ -butyl esters have been determined. Ultimate analyses of some of the hydrolysis products have been made. It is concluded that, concurrently with the hydrolysis that takes place on exposure to atmospheric moisture, water is absorbed by the hydrolytic products. Polymeric butyl titanates prepared by partial hydrolysis employing 1.0–1.5 mol. of water have the best film-forming properties. Considerable differences in hardness and adhesion have been found in butyl titanate films pigmented with samples of alumina known to differ in adsorptive activity.

III—The formulation of butyl titanate paints with leafing and some non-leafing pigments is described; they have been tested as protective coatings conferring heat-resistance and corrosion resistance, and some preliminary work on the preparation of anti-fouling paints is described. Non-leafing pigments such as metal oxides and Zn dust give satisfactory films, but are improved by the inclusion of a leafing pigment such as wet-ground mica. Small particle size of the pigment improves the hardness and the adhesion of the coating. The rapid settling of Zn dust in a paint is alleviated by the addition of a small amount of high-viscosity ethyl cellulose, but settling still occurs on prolonged standing.

J. W. D.

### PATENTS

**Inks for Printing on Polyethylene.** DuP. USP 2,639,998

Inks containing a hydrocarbon polymer or interpolymers, or one that has been chlorinated or chlorosulphonated, are suitable for printing on polyethylene. C. O. C.

**Drying Oils containing Dispersed Polymers.** Dow Chemical Co. BP 703,443

Dispersions of polymers, e.g. vinyl chloride–vinylidene chloride copolymers, in drying oils are prepared by adding an oil-soluble dispersing agent, e.g. castor oil, to the drying oil and stirring in a latex of the polymer to produce a mixture containing 10–35% polymer. Water may be removed by milling or by blowing at elevated temperature, and the dried films show increased tensile strength. E. C.

**Surface Coatings.** Dow Chemical Co. BP 703,444

One or more drying oils or drying-oil-modified alkyl resins are homogeneously mixed, e.g. with the aid of solvents or by hot milling, with a copolymer of vinyl chloride (35–75%) and vinylidene chloride (65–25%) which has a viscosity at 120°C. of 0.9–1.4 c.p. as a 2% solution in *o*-dichlorobenzene. The products are surface coating resins of improved resistance to water, alkali, and fire. E. C.

**Use of Zinc Oxide in Increasing the Viscosity of Heat-hardenable Polysiloxane Resins.** British Thomson-Houston Co. BP 707,143

Small amounts of  $ZnO$  increase the viscosity of liquid, heat-hardenable polysiloxane resins thus enabling heavier films to be applied in one operation either by dipping or spraying. The addition also increases the stability of the resin and yields films of superior craze resistance and flexibility. C. O. C.

**Water Plastic Paint.** Plaster Products (Greenhithe). BP 707,146

Coatings of a water plastic paint containing casein are rendered waterproof by brushing them when dry with a solution which insolubilises the casein, e.g. with 5% aq. potassium alum. C. O. C.

### Corrosion-preventive Coatings for Steel. Goodcliff-Eecen Industries Pty. BP 703,397

A corrosion-preventive coating for steel comprises a low-viscosity chlorinated rubber, an unsaponifiable plasticiser, e.g. chlorinated diphenyl (total 6-25%), a solvent, e.g. carbon tetrachloride (12%), a powdered heavy metal of size  $< 75 \mu$ , e.g. lead or zinc (82-63%), and an oxidation catalyst containing a metal of Group VIII, e.g. potassium osmate. E. C.

Colour Problems in Lithographic Printing (IX p. 256).

## VI—FIBRES; YARNS; FABRICS

### Interrelations of Chemical Science and the Textile Industry. D. W. Hill. *Chem. and Ind.*, 148-153 (6 Feb. 1954).

An account of the growth of fibre science—the elucidation of the chemical structure and molecular architecture of natural fibres, and the development of man-made fibres. The difficulties due to the effects of fibre structure upon the nature of the response to chemical reagents and to swelling media are outlined. The important contribution of paper chromatographic technique to the clarification of protein fibre structure is indicated. J. W. D.

### Physicist's Approach to Textile Technology. J. W. S. Hearle. *Text. Research J.*, 24, 123-130 (Feb. 1954).

A physicist's approach to problems in textile technology is put forward. The importance of the fibre and the expression of fibre properties are discussed. A. B.

### Static Electricity in Textiles. J. W. Ballou. *Text. Research J.*, 24, 146-155 (Feb. 1954).

The formation of static electricity in textiles and instruments for its measurement are discussed. The static properties of natural and synthetic fibres are detailed, and the physical effects of static charge discussed. A. B.

### Fundamental Questions concerning the Static Electrification of Textile Yarns—II. V. E. Gonsalves and B. J. van Dongeren. *Text. Research J.*, 24, 1-12 (Jan. 1954).

An apparatus is described for the determination of the static electrification of yarns by measurement of the charge produced when the yarn runs over a stainless-steel guide. The results of various experiments are detailed and discussed. A. B.

### Colour in Cotton. S. A. Simon and C. Harmon. *Text. Research J.*, 24, 12-16 (Jan. 1954).

The four main sources of colour in cotton are discussed, viz. dirt, fibre geometry, intrinsic colour, and extrinsic colour. It is concluded that the intrinsic colorants of cotton should not survive bleaching, and that the colour remaining after bleaching is extrinsic in nature, and is due to the action of fungi. A. B.

### Improvements of Lustre of Cotton. III—Correlation of Visual and Physical Aspects of Lustre. L. Fourt, R. M. Howorth, and M. B. Rutherford. *Text. Research J.*, 24, 61-66 (Jan. 1954).

The visual and physical aspects of lustre are discussed, and it is shown that the measurement of contrast ratio in yarns correlates with the average of numerous judgments by observers. A. B.

### Improvement of Lustre of Cotton. IV—Effect of Weaving Pattern on Lustre in a series of Bleached Cotton Fabrics. L. Fourt and M. B. Rutherford. *Text. Research J.*, 24, 67-72 (Jan. 1954).

The effects of variation of weaving pattern or construction on the lustre of a fabric are discussed. Sheerness also increases lustre, but this is counteracted by the use of finer yarns. A. B.

### Improvement of Lustre of Cotton. V—Fibre Shape in relation to Lustre. VI—Yarn Structure in relation to Lustre. L. Fourt, R. M. Howorth, M. B. Rutherford, and P. Streicher. *Text. Research J.*, 24, 156-163, 163-172 (Feb. 1954).

V—The effect of fibre shape on the lustre of cotton is discussed. The Fresnel's law effect is tested.

VI—The effects of fibre factors, mechanical processing factors, and whole-yarn factors on lustre are discussed. The results show that the three main means of improving lustre by yarn structure factors are lowering of twist, use of optimum plying twist, and singeing to remove free surface fibres. A. B.

### Relative Responsiveness of Certain Properties of Cotton Fibre to Microbial Action. P. B. Marsh, K. Bollenbacher, M. L. Butler, and G. V. Merola. *Text. Research J.*, 24, 31-38 (Jan. 1954).

Experiments to determine the relative responsiveness of certain fibre properties to the growth of fungi are described. In general, the aqueous-extract pH and the reducing-substance content of the incubated samples changed quickly and substantially, the former increasing and the latter decreasing. Various other changes in properties are described. A. B.

### Molecular Weight of $\alpha$ -Cellulose from Jute and Allied Long Fibres. H. Chatterjee, K. B. Pal, and P. B. Sarkar. *Text. Research J.*, 24, 43-52 (Jan. 1954).

Four methods commonly employed for the estimation of cellulose, with special reference to jute, have been critically studied by means of viscosity measurements of the  $\alpha$ -cellulose obtained with each in cuprammonium hydroxide. A formula is suggested to calculate the intrinsic viscosity, and hence molecular weight, from the relative viscosity of a cellulose solution at a single concentration. A. B.

### Hemp—The Effect of Retting. G. Centola. *Textil-Rund.*, 8, 649-656 (Dec. 1953).

In Italian, a summary in German is given *ibid.*, 8, 521 (Sept. 1953). A preliminary investigation leads to the following conclusions: retting does not take place evenly, the surviving pectin being localised; the pectin and lignin protect one another and are probably chemically linked; and retting appears to alter the composition of the pectin and possibly its connection with lignin. H. E. N.

### Microstructure of Rayons. E. Elöd. *Textil-Rund.*, 9, 13-19 (Jan. 1954).

Seven photomicrographs illustrate the skin structure of viscose (I) and cuprammonium rayon (II), demonstrated by oblique illumination, topochemical acetylation, phase-contrast, and by differential dyeing. Values recorded show that topochemical acetylation (from organic solvents) does not affect the D.P., breaking strength and extension at break of I. When treated with formaldehyde, the skin of I contains less formaldehyde, that of II more, than the corresponding core. Swelling of I in 70-30% aq. diglycol is slower than that of II but the final value is very similar. Removal of progressively more of the skin of I increases its rate of swelling. I with a thicker skin has better crease-resistance, but the improvement during a crease-resist treatment is greater with I with a thin skin and greatest with II. The final crease-resistance of treated, thick-skinned I is still better than that treated, thin-skinned I. H. E. N.

### Microcrystallite Size Determination by the Small-angle X-Ray Technique in Rayons. A. N. J. Heyn. *Text. Research J.*, 23, 782-787 (Nov. 1953).

The scattering of X-rays at small angles by various rayons of high orientation has been investigated at various degrees of swelling. Inflection points or maxima-minima are found to occur in the scattering curves at certain degrees of swelling, and this is explained by interparticle interference in the densely packed condition. The experimental conditions necessary for a reliable determination of particle size (diameter of microcrystallites) for these fibres are derived from the results obtained, and particle sizes determined by this method are given. A. B.

### The Structure of Viscose Rayon and Barriness in Dyeing. J. Meybeck, N. Iwanow, and R. Schneider. *Textil-Rund.*, 8, 589-602 (Nov. 1953).

The "differential dyeing method" (*Bull. Inst. text.*, 35, 51 (1952); 37, 7 (1953)) consists in preparing small lengths of fibres, 40-100  $\mu$  long, and dyeing these with a 2% soln. of Sky Blue FF for 2 min. at 20-22°C. The penetration of dye from the cut ends into the fibres is observed under a microscope and from measurements on more than 200 fibres a "penetration curve" (% fibres not penetrated to a distance  $\mu$  against distance  $\mu$ ) is obtained. The

penetration curve varies with the nature of the core of the sample and can be used as a method of control or test. Variations in the amount of stretch imparted by spinning have little effect on the penetration curve. Steaming reduces the penetration. Oxidation attacks the core but little as the penetration shows only minor alterations. The differential dyeing method is also used to investigate the effect of temp. on the rate of penetration. A new apparatus has been developed and the penetration of a single fibre can now be studied. Results are shown to be reproducible, the same fibre being repeatedly dyed and stripped. The rate of penetration decreases with increasing salt concn.

H. E. N.

#### Relation between External Shape and Internal Structure of Wool Fibres. E. H. Mercer. *Text. Research J.*, **24**, 39-43 (Jan. 1954).

Relations between the twisted bilateral structure of the cortex of the wool fibre and its external shape are pointed out. Both the crimped form and the elliptical cross-sectional shape follow the twisted internal structure. The geometrical relations of the crimp and ortho- and para-cortex are traced back to events occurring during keratinisation of the fibre.

A. B.

#### Structural Details of the Surface Layers of Wool. G. Lagermalm. *Text. Research J.*, **24**, 17-25 (Jan. 1954).

Electron-microscopical studies have been made on the surface layers of wool, separated from the rest of the fibre by several methods. It is shown that the epicuticle covers the fibre as a uniform layer, without being limited to the individual scales. The structural details of the exocuticle are discussed.

A. B.

#### Influence of Applied Forces on the Felting of Wool.

A. K. van der Vegt and G. J. Schuringa. *Text. Research J.*, **24**, 99-108 (Feb. 1954).

An apparatus is described for measuring the felting shrinkage of yarns at different force values. The course of shrinkage with reference to time can be satisfactorily defined by means of the initial shrinkage rate and the final limit of the shrinkage.

A. B.

#### Soluble Proteins of the Silk Gland of the Silkworm, *Bombyx mori*. E. H. Mercer. *Text. Research J.*, **24**, 135-145 (Feb. 1954).

The precursors of silk fibroin were extracted in sodium bicarbonate solution from the silk gland of the silk worm, *Bombyx mori*, and studied by ultracentrifugation.

A. B.

#### Casein Fibres. P.-A. Koch. *Textil-Rund.*, **9**, 25-28 (Jan. 1954).

An excellent table giving all the essential facts together with 72 references.

H. E. N.

#### Effect of Draw Ratio and Temperature on Electrical Conduction in Nylon Filaments. E. P. Sharman, S. P. Hersh, and D. J. Montgomery. *Text. Research J.*, **23**, 793-798 (Nov. 1953).

The electrical conduction in nylon has been studied as a function of draw ratio and temperature, and it has been found that the conductance depends upon the washing history. Using standard washing procedure, the conductivity at 30°C. was shown to decrease with increasing draw ratio to an extent greater than can be accounted for by the decreased moisture regain produced by drawing.

A. B.

#### Force-Temperature Behaviour of Nylon Filaments at Fixed Extensions. G. M. Bryant. *Text. Research J.*, **23**, 788-792 (Nov. 1953).

Force-temperature measurements on nylon filaments at fixed extensions in water have been related to the molecular mechanism of elasticity using the thermodynamic equations for elasticity. The behaviour of nylon 66 and nylon 6 was found to be the same, except that the former showed evidence of impending glasslike elasticity at the lowest temperature studied. Drawn fibres at low extensions were found to have an entropy force component larger than the total force.

A. B.

#### PATENTS

##### Retting Fibrous Plant Materials. O. Benedek.

BP 706,702

The time taken by biological retting of fibrous plant material is considerably shortened by pretreatment at

ordinary temperature with an aqueous solution containing 2-8% by wt. of an inorganic salt, other than phosphate, of an alkali metal or an alkaline earth metal, the solution being substantially neutral to phenolphthalein. Particularly suitable is  $\text{NaHCO}_3$ . Except when using such bicarbonates, it is advantageous to add up to 0.75 g. of HCl per l.

J. W. B.

##### Degumming Fibrous Plant Material. O. Benedek.

BP 706,699

Fibrous plant material, e.g. ramie or ramie refuse, is, prior to treatment with an aqueous solution or emulsion of a surface-active agent consisting of a sulphated fatty oil or fat and containing also a water soluble phosphate, heated in a dilute aqueous solution of a salt of an alkali metal or of an alkaline earth metal, the solution having a neutral reaction to phenolphthalein. In one example 0.5 kilo of ramie is boiled for 2.5 hr. in a 5% solution of  $\text{MgSO}_4$  or  $\text{NaHCO}_3$ .

J. W. B.

##### Treatment of Fibrous Plant Material for the Manufacture of Surgical Wadding. O. Benedek.

BP 706,701

Fibrous plant material, e.g. flax waste, which hitherto has been unsuitable for making surgical wadding because of the presence of non-cellulosic cementing substances, is treated at ordinary temperature with an aqueous solution of a water-soluble metallic salt having a neutral reaction to phenolphthalein, e.g.  $\text{MgSO}_4$  or  $\text{NaHCO}_3$ . A 5% solution for 72-120 hr. is suitable. Any remaining non-cellulosic material is removed by a further 2-3 hr. treatment in boiling 6-8% NaOH. The fibres can now be worked up into wadding in a manner similar to that used for cotton.

J. W. B.

##### Continuous Spinning of Viscose Rayon. N. V. Onderzoekingsinstituut Research.

BP 704,869

A rapid process for the production of viscose rayon by a continuous process is described. The regenerated fibre is treated with an aqueous solution containing an acid and an inorganic salt, thoroughly washed with water and then treated with salt-free acid solution till the xanthate ratio is ca. 0.10. After steaming to reduce the xanthate ratio to 0.003, the yarn can be desulphurised by washing in hot water.

W. G. C.

##### Esterifying Cellulose. Celanese Corp. of America.

BP 707,120

Cellulose can be esterified continuously by treating it first with a mixture of a lower aliphatic acid, water (20% on wt. acid) and an acid esterification catalyst (7.5-25.0% on wt. of acid) and then esterifying.

C. O. C.

##### Stabilising Polyamides to Heat Degradation. DuP.

USP 2,640,044

A mixture of a phosphorus compound hydrogen halide acid, or an alkali metal, alkaline earth metal or ammonium salt thereof when incorporated in synthetic linear polyamides imparts good resistance to degradation on heating.

C. O. C.

##### Increasing the Resilience and Resistance to Boiling Water of Polyvinyl Alcohol Fibres. DuP.

USP 2,636,803-4

The dry oriented fibres are heated in an anhydrous nonsolvent fluid medium to 1-30°C. below their softening point under such tension that they shrink 0-60% of their initial chain length until fibres are obtained which shrink < 10% of their length when immersed in water at 60°C. The fibres so set are treated in an aqueous bath at 1-15°C. below their softening point until they are saturated and swollen. They are dried in the swollen state and then treated in presence of an acetalisation catalyst with aminoaldehyde in which the HCO group is directly attached to a C atom forming part of a ring of > 4 atoms, until 25-75% of the OH groups in the fibre have combined with the aldehyde and the fibre has a softening point of > 175°C.

C. O. C.

##### Polyacrylonitrile Fibres. Courtaulds.

BP 706,262

Polyacrylonitrile fibres are produced by extruding polyacrylonitrile dissolved in an organic solvent which is soluble in  $\text{CCl}_4$  into a coagulating bulk consisting of or containing  $\text{CCl}_4$ .

Polyacrylonitrile dissolved in a water-miscible organic solvent is extruded into an aqueous coagulating bath containing < 20% by wt. of potassium acetate.

C. O. C.

#### Solvents for Acrylonitrile Polymers and Copolymers.

Koppers Co. BP 707,203  
 NN-Dimethylcarbamate esters of polyhydric alcohols (ester groups: C atoms in the alcohol::1:1-1.5) are solvents for acrylonitrile polymers and copolymers. The solution can be used for producing filaments, films, etc.

C. O. C.

#### Precipitating Liquids for Polyacrylonitrile Solutions.

Stockholms Superfosfat Fabriks. BP 706,154  
 Aromatic and hydroaromatic hydrocarbons are completely miscible with dimethyl sulphoxide, dimethyl formamide and  $\beta$ -aminopropionitrile. Aliphatic hydrocarbons except in special cases are not miscible with these solvents. This renders possible the use of cymene as a precipitating liquid for solutions of polyacrylonitrile in dimethyl sulphoxide and dimethyl formamide.

C. O. C.

#### Acrylonitrile-Vinyl Chloride Copolymers.

Union Carbide and Carbon Corp. BP 705,189  
 Copolymers containing 30-75% acrylonitrile and 70-25% vinyl chloride and having a specific viscosity between 0.15 and 0.7 are soluble in acetonitrile to form spinning solutions for making textile fibres, etc.

W. G. C.

#### Acrylonitrile Fibres of Good Dyeing Properties.

Chemstrand Corp. USP 2,639,969  
 Fibres of copolymers of acrylonitrile (75-98% by wt.) and of a vinyl ester of  $\alpha$ -chloroacetic acid may be rendered dyeable by treating them with ammonia, preferably with gaseous ammonia for 1 min. at 50-120°C. The treated fibres are readily dyed with acid dyes.

C. O. C.

#### Polysulphonamides.

Celanese Corp. of America. BP 705,427  
 Polysulphonamides having filament forming properties are obtained by treating a diamine in aqueous solution with a bis-sulphonyl chloride dissolved in a water-immiscible solvent and separating the polysulphonamide from the organic liquid phase.

W. G. C.

#### Improving the Stability to Light of Rubber-Sulphur Dioxide Fibres.

Courtaulds. BP 705,072  
 The resistance of polydiene-sulphur dioxide threads to photo-degradation is improved by extensive washing with alcohol to remove the free sulphur acid or other undesirable impurities.

W. G. C.

#### Butadiene Sulphur Dioxide Polymers.

Bataafsche Petroleum Maatschappij. BP 705,305  
 Filaments of improved mechanical properties are obtained by preparing the polybutadiene by emulsion polymerisation and controlling the molecular weight between 10,000 and 80,000.

W. G. C.

#### Crimping Yarns and Filaments.

N. Rosenstein and A. J. Rosenstein. BP 705,580  
 Continuous filaments or continuous filament yarns in tow or rope form are conveyed between limp sheathing members, such as limp endless bands, which pass through a suitable compression and distortion mechanism, in which the bands, and thus the enclosed filaments, are crimped. Setting in the crimped position is effected by steam and heat, after which the bands separate and the crimped filaments are discharged.

J. W. B.

#### Stable Detwisted Yarns and Fabrics containing them.

American Viscose Corp. USP 2,638,743  
 Yarns containing twist are coated with a film of an organic filament forming polyhydroxy compound soluble in water or weak alkali, e.g. polyvinyl alcohol, hydroxyethyl cellulose or sodium alginate. They are then detwisted, when the film serves to support them during processing until it is removed.

C. O. C.

#### Influence of the Method of Dyeing on the Wearing Properties of Wool (VIII p. 256).

[<sup>14</sup>C]-Cellulose from *Acetobacter Acetigenum* (XI p. 258). Depilating Animal Hides and Skins (XII p. 259).

## VII—DESIZING; SCOURING; CARBONISING; BLEACHING

### Soil Redeposition Prevention in Textile Cleaning Operations by Proteins and Other Polymeric Materials.

W. Fong and H. P. Lundgren. *Text. Research J.*, 23, 769-775 (Nov. 1953).  
 Certain proteins and other polymeric materials are highly effective in preventing redeposition of carbon black on cotton fabric in aqueous cleaning systems containing an alkylarylsulphonate detergent and alkaline builders. The efficiency of the various proteins tested in preventing soil redeposition could be correlated approximately with their proline content, the proline-rich proteins giving good to excellent whiteness retention. Several synthetic polymers were investigated, the uncharged types were more effective than the charged types.

A. B.

#### PATENTS

### Washing Raw Wool. Aktiebolaget Separator.

BP 705,805

Raw wool is washed with organic solvents by being continuously passed through a series of washing vats, the solvent being continuously led from each vat to the next in a direction opposite to that of the wool, and from the first vat back to the last. When passing from at least one vat to another the solvent is conducted through a purifier from which part of the washing agent is returned to the same vat from which it was discharged, and part to the vat preceding it or to the last vat in the case of solvent from the first vat. These partial flows are in purer condition with regard to sludge or wool-grease content than the solvent in the receiving vat. Economy in solvent is possible, and the system rapidly attains a state of equilibrium, thus making interruptions less of a nuisance.

J. W. B.

### Aftertreatment of Laundered Textile Goods.

FBy. BP 705,672

Textiles which have been laundered in an aqueous bath comprising a detergent containing a mineral auxiliary agent are aftertreated in a separate aqueous bath containing a lubricant softening agent, which obviates trouble from deposited metallic salts. In one example a viscose rayon fabric is washed 10 times at 80-94°C. with an aqueous detergent containing 5% by wt. paraffin sulphonate, 5% isododecylphenylsulphonate, 40% sodium carbonate and 4% sodium silicate, using 20:1 liquor ratio. After each washing it is rinsed and treated in an aqueous solution of 1 g./l. of a weakly sulphonated tallow, the liquor ratio being 20:1. A loss in strength of about 40% compares with a loss of about 90% where no softening baths are given. In some instances the strength is greatly increased.

J. W. B.

### Reaction between Chlorous Acid and Glucose—Quantitative Stoichiometry and Evaluation of Reagent Decomposition (III p. 246).

## VIII—DYEING

### Dyeing Properties of Vat Dyes.

J. Wegmann. *Textil-Rund.*, 8, 533-543 (Oct. 1953).  
 Graphs for 3 dyes of each of the 3 dyeing classes are given for the variation of exhaustion after 45 min. dyeing on both cotton and viscose rayon with (a) NaOH concn. (36°Be., 5-20 c.c./l.), (b) temp. (20-60°C.), and (c) salt concn. (0-40 g./l.). The differences in behaviour between the two fibres are very marked, there being no class III dyes as regards viscose rayon. Vat dyes are less temp. sensitive on cotton than on viscose. The curves are discussed in the light of practical experience.

H. E. N.

### Continuous Dyeing of Solubilised Vat Dyes.

C. T. Speiser. *Textil-Rund.*, 8, 602-613 (Nov. 1953).  
 A discussion of practical details, together with some new experimental material. Attention is drawn to the need for correctly running guide rollers. The cloth must leave the nip cleanly; if it follows one of the squeezing rollers, the liquor within it will be forced to the other side giving different depths of dyeing on back and face. The nip must be as near the surface of the trough as possible. In some machines, very suitable for light dyeings, the cloth does not pass through the trough, but receives liquor only

from the bottom roller of the mangle, the lower half of which is in the trough. Soaping is very important. Tables are given for the effect on dyeings of 21 Indigosol dyes (DH) of various times of soaping as measured by the change produced on further soaping. It appears that 15 sec. is sufficient for fully soaping some dyeings, whereas others require 15 min. With some dyes, a pale dyeing requires longer than a medium one. 6 vat dyes are tested for comparison. In general, anthraquinoid dyes need less soaping than indigoid dyes.

H. E. N.

#### The Effects of Soaping Vat-dyed Material. J. Müller. *Textil-Rund.*, 8, 622-624 (Nov. 1953).

Comments on Wegmann's work (cf. *J.S.D.C.*, 69, 267 (1953)). 3 g./l. indanthrone is reduced to its brown vat at 60°C. in presence of only 10 g./l. hydrosulphite simply by increasing the alkali content to 200 c.c. Boiling with water tends to reduce the azine-form, but presence of an oxidation catalyst, e.g. 5 mg./l. Co acetate, causes increase in formation of azine. When mercerising dyeings of indanthrone, azine formation can be prevented by adding glucose, hydroxylamine or hydrosulphite. Further comments and Wegmann's reply (*Textil-Rund.*, 8, 624-625 (Nov. 1953)) are polemical.

H. E. N.

#### Influence of the Method of Dyeing on the Wearing Properties of Wool. P. Fink. *Textil-Rund.*, 8, 279-302 (June); 346-368 (July); 543-557 (Oct.); 613-622 (Nov. 1953).

The literature is surveyed and much new experimental material is presented. Assessment was by the damage number, a microscopic-microchemical test, using the following reactions with these weights: alteration of fibre surface 15; coloration with ammoniacal silver nitrate soln. 10; with dyes 10; with Pauly reagent 10; Allwörden reaction 10; pH value 5. Full details are given and accuracy is judged to be 5-10%. Other methods for evaluating damage are reviewed and employed: xanthoproteic reaction, swelling with a soln. of 10 g. KOH in 25 c.c. ethylene diamine, alkali solubility, bichromate number, acid and alkali binding capacity, total sulphur and sulphide-sulphur content, chrome content, water content, breaking length and elongation at break. The results of tests are considered statistically. It is shown that the absorption of bichrome follows the laws of Freundlich and Langmuir. In afterchroming, the chromic acid largely replaces the acid absorbed in dyeing, the chromic acid is subsequently reduced, leaving the wool in a less acid condition than after acid dyeing. Combination with chrome does reduce the reactivity of wool. Boiling acid attacks the disulphide bond, but the action of acid is dominated by the acid combining capacity of the wool. No dyeing process leads to definite attack on the sulphur in wool. Long boiling in acid liquors is less bearable than a mild alkaline treatment at moderate temp. (vat dyeing), but longer alkaline treatments clearly alter the wool. Wool dyed with a selection of dyes of various classes is fully examined for the changes produced in its resistance to exposure to both sunlight and ultra-violet radiation (I); washing (II); steam under slight pressure (III); and acid (IV). The chemical and mechanical methods of evaluation correlate well, but slight chemical changes can be detected chemically without appreciably altering the mechanical properties. Of the treatments examined, I has by far the most serious effect. Chroming protects against I and its action seems independent of the degree of chroming or of the fastness of the dye. Dyeing with indigoid or solubilised vat dyes also protects against I, the increases in the probable life of the fabric being up to 100%. The hue of the dye is without effect on the action of I. II and IV produce only minor changes, vat-dyed wool being affected most. III, and II following on I, have little effect on the chemical properties but more seriously reduce the mechanical ones. Protection against II, III, and IV by dyeing is negligible or absent.

H. E. N.

#### Methods of Research in Textile Chemistry. R. Haller. *Textil-Rund.*, 9, 1-13 (Jan. 1954).

Historical review, 43 references.

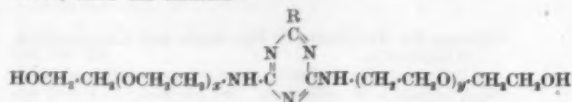
H. E. N.

#### PATENTS

#### Dyeing Nitrogenous Fibres with Metallised Azo Dyes. American Cyanamid Co. *USP* 2,638,404

When dyeing with metallised azo dyes in strong acid baths, level dyeing without seum formation such as is

caused by cationic active surface agents, is obtained by addition of polyalkylene oxide derivatives of 2-alkyl guanamines, which, in the case of ethylene oxide derivatives, have the formula—



(R = aliphatic hydrocarbon radical of > 6 C; x + y = 9-98).

C. O. C.

#### Developing Colorations produced with the Tetra-sulphuric Acid Leuco-Ester of Tetrahydro-1:2:2':1'-dianthraquinoneazaine. DH. *BP* 707,132

Satisfactory dyeings and prints are obtained by acid oxidation in steam development processes in presence as buffer of a sulphonic or carboxylic acid of a mono- or dihydroxynaphthalene. Addition of such a buffer or its salt to the dyebath or printing paste makes possible variation in the proportion of the substances necessary for development without any change in hue of the developed dyeing or print, and they are almost insensible to variations in the steaming conditions.

C. O. C.

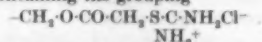
#### Treating Acrylonitrile Fibres with Zinc Chloride to Increase their Affinity for Acid Dyes. Chemstrand Corpn. *USP* 2,636,802

Treating acrylonitrile fibres with aq.  $\text{ZnCl}_2$  increases their affinity for acid dyes. The nature of the action involved is not known as treatment with other solvents under comparable conditions does not produce this effect. It is not a permanent alteration for the treated fibres may have all the  $\text{ZnCl}_2$  removed by washing, but so long as they remain moist the increased affinity for acid dyes remains but drying removes this affinity.

C. O. C.

#### Thiourea as Assistant in Dyeing Acrylonitrile Polymers with Acid Dyes. Chemstrand Corpn. *USP* 2,637,620

Addition of thiourea to the bath when dyeing acrylonitrile polymers with acid dyes results in vivid dyeings fast to washing and dry cleaning. The effect is probably caused by the thiourea and the chlorine of the polymer forming a salt containing the grouping



after which anionic exchange occurs between the ionic chlorine and the dye anion.

C. O. C.

#### Natural Dyewoods and their Application—Logwood (IV p. 249).

#### The Structure of Viscose Rayon and Bariness in Dyeing (VI p. 253).

#### Esterifying Cellulose (VI p. 254).

#### Pigment-Resin Emulsions for Colouring Textiles (IX p. 257).

#### Reaction between certain Acid Dyes and Size Precipitate (XI p. 258).

#### Aggregation Tendencies of Acid Wool Dyes in Saline Solutions (XV p. 260).

## IX—PRINTING

#### Mechanised Screen Printing. A. Franken. *Textil-Rund.*, 8, 656-663 (Dec. 1953).

Various machines are described, including one for sticking down the piece, one for washing down the table, several automatic screen printing machines, and a double-spiral steamer.

H. E. N.

#### Colour Problems in Lithographic Printing. J. H. Brittain and A. D. Lott. *J. Oil and Col. Chem. Assoc.*, 36, 601-618 (Nov. 1953).

The colour produced by a lithographic printing ink depends principally upon its film thickness (modern machines normally lay a film ~ 1-3  $\mu$ . thick, whereas full opacity requires a thickness of ~ 50  $\mu$ .), the reflectance of the background, and several other factors. Methods now in general use for evaluating the optical properties of these inks are inadequate. Diffuse reflectances of two inks (red and yellow) laid down over several neutral backgrounds at a range of film thicknesses followed certain patterns. Results are tabulated for the trichromatic

coefficients and luminance factors *versus* film thickness for each ink laid upon black, white, and three neutral grey backgrounds; these are discussed and some are plotted. Increases in the thinnest films produce large increases in saturation; as film thickness increases the changes in saturation become smaller. There is at the same time change in the dominant wavelength with film thickness, towards that of the infinitely thick film. The luminance factor depends also upon film thickness and background lightness, and that relationship differs for the two inks; this can only be (and is) satisfactorily shown by a locus in the colour solid. From the above curves the coefficients of absorption and scattering have been calc. for both inks by application of the Kubelka-Munk equation. Reflectances predicted from these data compare well with measured values and indicate that this relationship may be of practical value in the study of lithographic printing inks. J. W. D.

## PATENTS

**Pigment-Resin Emulsions for Colouring Textiles.**

L. Auer. USP 2,637,621

Use as binder of a polyester formed by drying oil fatty acids esterified with the condensates of epihalogenohydrins or dihalogenohydrins with polynuclear phenols, e.g. bisphenol, results in good fastness to rubbing. C. O. C.

**Oil-in-water Emulsions as Printing Pastes.**

L. Auer. USP 2,637,705

Oil-in-water emulsions formed from (1) high viscosity methyl cellulose or carboxymethyl cellulose, (2) water, and (3) water-immiscible solvents, e.g. aliphatic or aromatic hydrocarbons, are particularly suitable as extenders of oil-in-water emulsion pigment concentrates and as carriers for vat dyes. Their use gives good colour yield, enables the prints to be stored for longer periods before ageing and prevents any trouble in removal of "gums" after ageing. They can also be used to print acid, direct, solubilised vat and azoic dyes. C. O. C.

**Aqueous Dispersions of Organic Pigments.**

L. Auer. USP 2,637,711

Dispersions of high colour yield and tinting power are produced by using fatty alcohol sulphates as dispersing and wetting agents for organic pigments using, preferably, 15–20% on the dry weight of the pigments. These agents work well alone but their action is improved by presence of smaller amounts of sodium alkyl-naphthalene sulphonates and further improvement in the degree of deflocculation of the pigments is obtained by addition of protective colloids, e.g. casein or methyl cellulose. They are used for obtaining textile prints which are fast to washing. C. O. C.

**Multi-colour Printing.**

A. I. Sark. BP 707,137

A roller or block made up of a mosaic of differently coloured pigments imparts at one impression a coloured pattern to a matt non-absorbent roller which in turn imparts the pattern to the material to be printed. C. O. C.

**Multi-colour Printing.**

Chromostyle. BP 707,381

Multi-coloured designs obtained by use of a single multi-coloured printing roller or block are water-resistant if in the coloured compositions used there is incorporated polyvinyl chloride or acetate, synthetic resins, rubber, combinations of vinyl resins or latices with nitrile-type rubbers, cellulose nitrate, or mineral wax in absence of any substance insoluble in the solvent. C. O. C.

**Recent Progress in Sensitising Dyes** (IV p. 248).Brown, etc. *Azoic Dyeings and Prints* (IV p. 250).

Developing Colorations produced with the Tetrasulphuric Leuco-Ester of Tetrahydro-1:2:2':1'-dianthraquinone-azine (VIII p. 256).

Melamine Transfer (XIII p. 259).

**X—SIZING AND FINISHING****Preparation of Microbiologically Resistant Wool by means of Chemical Modification. II—****Paper-chromatographic Investigation.**H. Zahn. *Text. Research J.*, 24, 26–31 (Jan. 1954).

The chemically modified wool samples described in Part I (Zahn and Wilhelm, *ibid.*, 23, 604 (1953)) have been investigated by paper chromatography. It is pointed out that in some cases the paper-chromatographic method of

investigation offers the sole means of analysis of chemically modified wool. Its significance for the chemistry of wool research has been demonstrated in the formation of lantionine in wool treated with acetone and bicarbonate. A. B.

**Reaction of Wool with N-Bromoacetamide and N-Bromosuccinimide.**C. Earland and D. Raven. *Text. Research J.*, 24, 108–112 (Feb. 1954).

The reaction between wool and organic bromamines has been investigated to determine whether they react as simple brominating agents or whether the reaction is more complicated, as with chloramines. It is shown that certain bromamines would appear to constitute a useful class of anti-felting agent. A. B.

**Moisture Relations of Knit Fabrics of Wool and Several Synthetic Fibres and Blends.**M. J. Coplan. *Text. Research J.*, 23, 897–916 (Dec. 1953).

Study of a variety of fibres in similar knit structures to determine the effect of inherent fibre properties on speed of drying showed that the total drying time under household drying conditions depends upon the ratio of total moisture uptake to peripheral surface area of fabric. The drying mechanism depends upon vapour diffusion from the atmospheric "well" within the fabric to its periphery, and thence through a boundary film, where mass-energy transfer occurs. A. B.

**Polymerisation of  $\beta$ -Propiolactone in Wool.**W. G. Rose and H. P. Lundgren. *Text. Research J.*, 23, 930–936 (Dec. 1953).

The reaction of propiolactone with wool has been studied with the purpose of increasing the rate of reaction within the fibre, while still retaining the enhanced felting qualities of the modified wool. It was found that presence of water in low concentration, added either to the wool or to the propiolactone in carbon tetrachloride, greatly accelerated this reaction. Excess water reduced the reaction, presumably because of lactone hydrolysis. The reaction was also investigated when alcohols were used as solvents. A. B.

## PATENTS

**Increasing the Tensile Strength of Yarns.**

Specialities Development Co. USP 2,637,658–9

The yarn is impregnated with a bonding agent while being rolled at several points along its length to slightly loosen or untwist the individual fibres and the bonding agent set while the yarn is stretched.

**Imparting Lustre and/or Firmness to Fabrics.**

William Franklin &amp; Son. BP 707,322

The fabric containing 10–50% of its dry weight of water is drawn under tension over a stationary, convex surface heated to  $>100^{\circ}\text{C}$ . The tension is imparted by leading the fabric from the surface more quickly than it is fed to it. This imparts high lustre without glazing or flattening of the selvages. C. O. C.

**Fabrics from Adhesively Bonded Fibres.**

Johnson and Johnson (Gt. Britain). BP 705,057

To a layer of carded web fibres which are easily wetted, e.g. bleached cotton or regenerated cellulose, is applied a similar layer of less wettable fibres such as unbleached cotton. The two are cemented by printing a lattice pattern of suitable adhesive which penetrates the first web easily and the other slightly but which is adequate to hold them together. Not being thoroughly wetted, the less-wettable layer takes the form of a soft nap bonded to the first web. It is suggested that a fabric of this sort may be of particular value as a base for wallpaper. The fabric is bonded by the wettable side to the wall, e.g. with a starch paste, and wallpaper is applied in the usual way to the nap side. It can subsequently be stripped off very easily. J. W. B.

**Milled Textile Fabrics.**

H. Hassan. BP 705,055

A milled textile fabric is made by felting cloth which is woven from yarns containing wool fibres in the weft and from yarns of artificial fibres which decrease in strength on wetting, e.g. viscose rayon, reinforced by yarns of vegetable fibres which increase in strength on wetting, e.g. cotton, in the warp. The warp threads may be alternately one type and the other, or may comprise the two types twisted together. It is possible to mill such a fabric so that no undue strain is put on the weak warp components,

particularly if the first stage of milling is gentle. Production cost is reduced and a cleaner faced melton cloth can be obtained as a result of partly eliminating fine cotton fibre ends.

J. W. B.

#### Finish for Cotton Glove Fabric. R. N. Russell.

USP 2,637,662

Cotton glove fabric is given a suede-like, perspiration-proof finish by impregnating it with a composition containing lanolin (4%), mineral oil U.S. Pharmacopoeia No. 8090 (6-16), stearic acid (2-8), propylene glycol (9-12), triethanolamine (1.25-2.0), terpineol-almond extract (0.1-0.2), quince seed gum (1.5-2.5) and water (to bring to 100). The finish resists repeated cleanings.

C. O. C.

#### Coating and Impregnating Textiles, Paper, etc. with Olefin-Sulphur Dioxide Resins. Phillips Petroleum Co.

USP 2,637,664

Smooth, tightly-bonded resinous coatings are obtained by use of an olefin-sulphur dioxide latex at pH 6-9.

C. O. C.

#### Non-woven Soft-surface Floor Covering. Bigelow-Sanford Carpet Co.

USP 2,638,959

A flexible backing sheet coated with a thermoresponsive adhesive is advanced intermittently and during the stoppages bights of the surface material are pressed in rows across the width of the backing into the soft or softened adhesive. The adhesive is responsive to heating by dielectric losses produced by a high frequency electric field simultaneously with the pressing of the surface material into the adhesive, thus producing firm bonding between that material and the backing.

USP 2,639,250

Any suitable means of heating the adhesive may be used.

C. O. C.

Stable Detwisted Yarns and Fabrics containing them (VI p. 255).

Methods of Research in Textile Chemistry (VIII p. 256).

## XI—PAPER AND OTHER CELLULOSIC PRODUCTS

**Physical Characteristics and Chemical Composition of  $\alpha$ -,  $\beta$ - and  $\gamma$ -Celluloses. I—X-Ray Diffraction Analyses.** K. J. Björkqvist, L. Jørgensen, and A. Wallmark. *Svensk Papperstidning*, 57, 113-118 (28 Feb. 1954).

Analyses of several alkali-soluble fractions from different pulps and from hydrolysed spruce holocellulose showed X-ray diffraction methods to be too insensitive for quantitative estimation of amorphous material mixed with cellulose II. The  $\beta$ -cellulose from sulphite rayon pulp has a cellulose II diffraction pattern, whilst the other  $\beta$ -celluloses contained varying amounts of amorphous material. Part of the  $\gamma$ -cellulose from sulphite rayon pulp has possibly a crystalline pattern, but the other  $\gamma$ -celluloses were amorphous. X-Ray diffraction analysis of alkali-soluble fractions cannot be used as definite proof of the nature of these heterogeneous materials.

S. V. S.

**Wet-strength Paper and its Production by Beater Addition of a Copolymer of Butadiene and Acrylonitrile.** K. G. Larsson and G. Thurne. *Svensk Papperstidning*, 57, 73-77 (15 Feb. 1954).

Nitrile rubber can be used for wet-strengthening paper instead of the usual resins, the coagulation procedure being simple but the latex-treated papers requiring heating for some minutes at 200°C. Such heating also improves the wet strength of unimpregnated paper considerably.

S. V. S.

**Synthesis and Reactions of (3-Methoxy-4-hydroxyphenyl)-glycerol (Guaiacylglycerol). III—Sulphonation and "Ethanolysis".** E. Adler and S. Yllner. *Svensk Papperstidning*, 57, 78-82 (15 Feb. 1954).

As with other *p*-hydroxybenzyl alcohols, guaiacylglycerol is rapidly sulphonated by sulphite solutions over a pH range of 1.5-9. On heating guaiacylglycerol with ethanolic HCl,  $\alpha$ -ethoxypropionguaiacone, methyl vanilloyl ketone, and guaiacylacetone are formed. These and some other lignin model reactions of guaiacylglycerol, veratryl

glycerol, and the  $\beta$ -guaiacyl ether of veratryl glycerol are summarised, and the possible occurrence of guaiacylglycerol  $\beta$ -aryl ether linkages in lignin is discussed.

S. V. S.

**Reaction between certain Acid Dyes and Size Precipitate.** A. J. M. Draper. *Proc. Tech. Sectn. British Paper and Board Makers' Assoc.*, 34, 497-507 (Dec. 1953).

Certain acid dyes have been shown to possess an affinity for rosin size-alum precipitate. An adsorption isotherm has been plotted for Disulphine Blue FFN, and it has been shown that differences in affinity can be demonstrated by desorption of dye with sodium sulphate and desorption of one dye with another. The ratio  $[D]_s/[D]_f$  (the respective concentrations of dye on the fibre and in solution) was thought to be a possible measure of relative affinities and was estimated for Disulphine Blue FFN and Crocein Scarlet 3B, but did not appear to be constant for any particular dye. The difference in affinity of two dyes for size precipitate may be used as the basis for separating them from a mixture.

S. V. S.

**Chromatographic Fractionation of Wood Carbohydrates.** R. R. Bridges. *Regional Conclave of the American Chemical Society. Anal. Chem.*, 25, 1935 (Dec. 1953).

A complex mixture of water-sol. carbohydrates resulting from the hydrolytic degradation of wood hemicelluloses in the production of fibre by the Masonite process has been partially resolved into 20 individual simple sugars and oligosaccharides by the application of a combination of the charcoal column chromatography of Whistler and Durso (*J. Amer. Chem. Soc.*, 72, 677 (1950)) and large sheet paper chromatography. Ten of the compounds are known and have been identified, but the remainder have not been definitely identified except with respect to their size and the simple sugar units of which they are composed, both of which have been obtained from paper chromatographic evidence. The simple sugars isolated are xylose, arabinose, glucose, galactose, and mannose, and the oligosaccharides are made up of units of these sugars (except arabinose) arranged either in polymers of different sugar units or the same sugar units ranging in size from 2 to 6 units. The unidentified components are oligosaccharides larger than six units, these not being readily separated by the methods employed.

J. W. D.

**[<sup>14</sup>C]-Cellulose from *Acetobacter acetigenum*.** E. J. Bourne and H. Weigol. *Chem. and Ind.*, 132 (30 Jan. 1954).

*Acetobacter acetigenum* (T. K. Walker's strain) was grown at 29°C. for 27 days at pH 5.8 on a medium containing  $\text{NH}_4^+$ ,  $\text{K}^+$ ,  $\text{Mg}^{2+}$ ,  $\text{Ca}^{2+}$ ,  $\text{Fe}^{2+}$ ,  $\text{Zn}^{2+}$ ,  $\text{PO}_4^{3-}$ ,  $\text{SO}_4^{2-}$ ,  $\text{CO}_3^{2-}$ , DL-alanine, L-asparagine, DL-valine, L-leucine, DL-iso-leucine, L-glutamic acid, *p*-aminobenzoic acid, nicotinic acid, calcium DL-pantothenate, riboflavin, biotin, *D*-glucose, ammonium lactate, and DL-(carboxy-<sup>14</sup>C)-lactic acid. The [<sup>14</sup>C]-cellulose (I) produced yielded, on purification, hydrolysis, and paper chromatography, only glucose. The distribution of radioactivity in (I) was determined by converting each individual carbon position into  $\text{BaCO}_3$ . The results, which are tabulated, show that the glucose carbon chain is symmetrically labelled, suggesting that the glucose units result from the fusion of two three-carbon fragments; further evidence for this is currently being sought.

J. W. D.

#### PATENTS

**Acid-resistant Wet-strength Paper.** American Cyanamid Co. USP 2,639,242

The paper stock is treated with the water-soluble condensate of a phenol, formaldehyde and the salt of an aminocarboxylic acid.

C. O. C.

**Compositions for Making Paper Thermosensitive.** Product Development Laboratories. USP 2,637,657

A mixture of a heavy metal salt which decomposes on heating to yield a coloured product, e.g. lead formate, and a compound which decomposes at a lower temperature, e.g. mercuric oxalate is applied to paper for use with a heated tracing device. The heat given off by the decomposition of the second compound added to that applied by the secondary instrument results in effective decomposition of the heavy metal salt.

C. O. C.

### Cyclo-Rubber for Application to Regenerated Cellulose Sheets. Dainihon Cellophane Kabushikigaisha.

BP 705,712

Raw rubber in benzene solution is reacted with stannic chloride in aqueous solution in the presence, or before the addition, of at least four mols. of caustic soda, caustic potash or ammonia per mol. of stannic chloride. The reactants may be stirred while the alkali is added to form an emulsion which is steam-distilled. The cyclo-rubber solution produced is applied to the surface of regenerated cellulose sheets to increase their age resistance.

S. V. S.

## XII—LEATHER; FURS; OTHER PROTEIN MATERIALS

### PATENT

#### Depilating Animal Hides and Skins. H. Zalman.

BP 706,746

A "paint" composition, of the type used for dewooling sheepskins, comprises an aqueous suspension having HS ions in solution, these being provided by a sulphide or hydrosulphide of Ca, Ba, Sr, Mg, or Al. Free and reserve OH ions are provided by a Ca, Ba, or Sr hydroxide. The composition must be free of monovalent cations. Ranges of OH<sup>-</sup> and SH<sup>-</sup> concentration are defined diagrammatically. Control of pH down to the minimum necessary for efficient depilation is effected along with an economic maximum of SH<sup>-</sup> concentration.

J. W. B.

Alkali-Lignin Tanning Agent (III p. 247).

## XIII—RUBBER; RESINS; PLASTICS

### PATENTS

#### Coloured Polymerisation Products. Chemische Werke Huls.

BP 706,104

When monomeric vinyl compounds are polymerised in an aqueous suspension of powdered solids insoluble in the vinyl compounds and in water, polymerisation being carried out by simple stirring in presence of a polymerising catalyst and in absence of emulsifying agents, coloured products can be obtained by carrying out the reaction in the presence of pastes obtained by grinding pigments with oily esters of higher aliphatic carboxylic acids. Such pretreated pigments even when of much higher s.g. than the vinyl compounds do not settle out during the polymerisation.

C. O. C.

#### Melamine Transfer. Di-Noc Co. USP 2,639,253

Hard-surfaced laminates are prepared by using several layers of resinous film-forming materials, one of which is a decorative layer preferably a dry-stripping transfer of the type described in USP 2,606,853. This avoids use of carrier sheets and also yields designs having no fuzziness.

C. O. C.

Use of Zinc Oxide in Increasing the Viscosity of Heat-hardenable Polysiloxane Resins (V p. 252).

Solvents for Acrylonitrile Polymers and Copolymers (VI p. 255).

Cyclo-Rubber for Application to Regenerated Cellulosic Sheets (XI above).

## XIV—ANALYSIS; TESTING; APPARATUS

**Colorimetric Method for Quantitative Micro-determination of Quaternary Ammonium Compounds—Losses of Quaternary Ammonium Compounds caused by Glass Adsorption and Concentration in the Foam.** J. Fogh, P. O. H. Rasmussen, and K. Skadhauge. *Anal. Chem.*, **26**, 392-395 (Feb. 1954).

Cetylpyridinium chloride (I) may be determined in concn. ranging from 0 to 25 µg./ml. by its formation of a blue complex with bromocresol purple, the intensity of which is measured spectrophotometrically at 620 mµ., and at a pH < 8.2. The results are unaffected by variations of temp. within the range 18-35°C., and by the presence of Ca<sup>2+</sup>, Mg<sup>2+</sup>, Fe<sup>3+</sup>, Fe<sup>2+</sup>, and Cu<sup>2+</sup> up to concn.

~1%. The quaternary ammonium compound is strongly adsorbed on to glass surfaces, especially if these are scratched or marred, losses of I due to this adsorption have been found to vary from 0 to 70% of an initial concn. of 1/50,000; this can be minimised by treating the interior surfaces of all glassware used in the determination with polymethacrylic ester (Plexiglas). On shaking, the foam phase tends to increase its concn. of I at the expense of the liquid phase. The method may be used to establish standard curves for the determination of other quaternary ammonium compounds.

J. W. D.

### Spectrophotometric Titrations with Ethylenediaminetetra-acetic Acid (II)—Determination of Magnesium, Calcium, Zinc, Cadmium, Titanium, and Zirconium. P. B. Sweetser and C. E. Bricker. *Anal. Chem.*, **26**, 195-199 (Jan. 1954).

Procedures are described in which the end-points of titrations in which ethylenediaminetetra-acetic acid (I) is used as indicator, are detected spectrophotometrically in the ultra-violet region (at wavelengths depending upon the metal ion being determined), the metals being held either singly or in admixture, in ammonia-ammonium chloride buffered soln. Soln. of (I) as dil. as 0.001 M. may be used even when the total vol. of soln. being titrated is 100 ml.

J. W. D.

### Use of Infrared Radiation for Detection of Colourless Substances on Paper Chromatograms. D. R. Kalkwarf and A. A. Frost. *Anal. Chem.*, **26**, 191-195 (Jan. 1954).

The emission or transmission of infra red radiation by a developed paper chromatogram and that by a plain filter paper, are compared by an instrument in which they are scanned at a steady rate. A motor-driven chopping wheel allows the radiation to pass from each in turn at a rate of 20 cycles per sec. The intensity amplitude of the pulsating beam of radiation is equal to the difference in the intensities of radiation from the two strips. The alternating intensity of the radiation is registered by a thermistor-type bolometer, the resulting alternating electric signal being amplified, rectified and recorded. The apparatus is fully described and there are sketches. There is a detailed discussion of recordings obtained from developed chromatograms derived from mixtures of several substances including phthalic acid, naphthalene β-sulphonic acid, oxalic acid, and dextrose, the recorded traces themselves being shown. The method is relatively insensitive in detecting sugars, possibly on account of their close chemical relationship to cellulose.

J. W. D.

### Determination of Phenols in Waste Waters by Ultra-violet Absorption. L. J. Schmauch and H. M. Grubb. *Anal. Chem.*, **26**, 308-311 (Feb. 1954).

The ultraviolet spectra of monohydric monocyclic phenols undergo a bathochromic shift with increase in pH. Oil is removed from the water sample by extraction with CCl<sub>4</sub> at pH 12, and the phenols are removed by extraction with tributyl phosphate at pH 5. The resulting concentrate is divided into two portions, one of which is returned to pH 12. Comparison at 3010 Å. of the two portions gives a measure of the phenols, an absorbance of 0.015 per cm. corresponding to 0.01 p.p.m. of phenols. Naphthols and dihydric phenols interfere, but thiophenol, basic nitrogen compounds, carboxylic acids, and sulphonic acids have negligible effect. Experimental details are given in full. Although the determination requires ca. 1½ hr. to complete, 1 hr. of this time is occupied by extractions which can proceed unattended.

J. W. D.

### 2-Acetoacetyl-4-methylphenol as a Reagent for Primary Amines. J. B. Harborne and A. S. Weaving. *J.C.S.*, 1157-1158 (March 1954).

2-Acetoacetyl-4-methylphenol (I) does not react with primary aromatic amines at room temp., whereas *o*-, *m*-, and *p*-phenylenediamines react slowly, or more rapidly on heating. On paper chromatograms, the latter, after spraying with the reagent, appear as bright yellow spots, which can be readily distinguished from the corresponding spots given by primary aliphatic amines. A number of amines are characterised by reaction with I.

H. H. H.

**Determinations of Thiosulphate and Nitrite.** R. H. Pierson. *Anal. Chem.*, **26**, 315-320 (Feb. 1954).

A procedure is described whereby thiosulphate and nitrite may be determined in ammoniacal soln. when both ions are simultaneously present. The soln. is treated with  $\text{AgNO}_3$  under very carefully controlled pH conditions (a pH meter is used);  $\text{S}_2\text{O}_3^{2-}$  yields  $\text{Ag}_2\text{S}$  quant., and this is filtered off and determined by the Volhard method. Nitrite, which is unaffected by the above treatment, is determined in the filtrate by cerate oxidimetry. It is essential that  $\text{S}^{2-}$  and  $\text{SO}_3^{2-}$  are absent. Methods are given for the determination of  $\text{S}_2\text{O}_3^{2-}$  in soln. of  $(\text{NH}_4)_2\text{S}_x$  in the absence of  $\text{NO}_2^-$  and  $\text{SO}_3^{2-}$ , and of  $\text{SO}_3^{2-}$  in presence of  $\text{S}_2\text{O}_3^{2-}$  and  $\text{NO}_2^-$ . J. W. D.

**Chemisorption in the Chromatographic Separation of Organic Isomers and Homologues.** J. L. Forstner and L. B. Rogers. Pittsburgh Conference on Analytical Chemistry and Applied Spectroscopy. *Anal. Chem.*, **26**, 426 (Feb. 1954).

The separation of 2- and 4-nitroso-1-naphthols is improved by chelate formation, whilst co-ordination complex formation similarly enhances the separability of lower aliphatic amines; the improvements are especially marked when sorbents are used which possess little or no physical sorption. The effect of different cations on the separations of amines can be correlated with the relative strengths of the complexes. There is evidence that a similar relationship may obtain in separations involving chelates. J. W. D.

**Applications of Statistical Methods to Exposure Trials. I—Analysis of Exposure Data.** H. R. Touchin. *J. Oil and Col. Chem. Assoc.*, **36**, 709-720 (Dec. 1953).

The use of statistical methods renders exposure trials more efficient, since more information may be obtained from a given amount of work, and the extent of the errors involved in any experiment is determinable. Trials must be suitably designed to yield profitably to statistical analysis. Correlations between variables may be studied, and by an extension of this principle data may be combined into single values. The methods are limited to some extent, but not invalidated, by the difficulty of assessing the breakdown of exposed panels other than by subjective means. Numerous examples are given illustrating that conclusions drawn from visual inspection are not always justified, certain advantages accruing from the use of statistical methods. An example is included to illustrate the use of the correlation coefficient in relating accelerated and normal exposure methods. J. W. D.

**Assay of Certain Organic Materials by Dichromate Oxidation.** N. F. Schulz. *Anal. Chem.*, **25**, 1762-1764 (Nov. 1953).

Starches may be determined in soln. containing  $\geq 0.2$  g./l. by heating with dichromate- $\text{H}_2\text{SO}_4$  reagent for 2 hr. at  $100^\circ\text{C}$ . (or for 30 min. at  $120^\circ\text{C}$ . in an autoclave), unused excess dichromate being titrated with ferrous ammonium sulphate in presence of phosphoric acid; alternatively a spectrophotometric finish may be applied, the absorbance at 422-440 m $\mu$ . being measured. In both cases standardisation against a known soln. is essential. The extent of oxidation of 7 starch products by this method is indicated. Laurylamine acetate or sodium oleate may be similarly determined by modifying the procedure (higher concn. of acid is required). Procedures for determining starch and/or laurylamine acetate are detailed. J. W. D.

**Determination of Dextran with Anthrone.** T. A. Scott and E. H. Melvin. *Anal. Chem.*, **25**, 1656-1661 (Nov. 1953).

A standardised method has been worked out for determining dextran by means of the colour developed when its soln. is heated with anthrone in  $\text{H}_2\text{SO}_4$ . The work leading to the establishment of optimum reaction conditions is outlined. After development of the colour the absorbance is measured at 625 m $\mu$ . The method is detailed. Numerous sources of error have been studied, this work being described, the most probable being found to be variation in the blank. Methods are discussed

whereby the interference of other carbohydrates may be determined; the extent of interference of 19 substances, organic and inorganic, is indicated. The standard deviation, in absorbance units, is 0.0029, corresponding to 0.48% at an absorbance level of 0.600. J. W. D.

**Rapid Accurate Determination of Cellulose with the Dichromate Heat-of-dilution Method.** H. F. Launer and Y. Tomimatsu. *Anal. Chem.*, **25**, 1769-1770 (Nov. 1953).

Results obtained by the heat of dilution method are compared with those given by a method requiring external heating (Kettering and Conrad, *Ind. Eng. Chem. Anal. Ed.*, **14**, 432 (1942)) for identical samples of celluloses, starch, and glucose, with identical reagents. A combination of the two methods is embodied in the TAPPI and ASTM standard procedures, the heat of dilution method being used to determine the alkali-soluble fraction. The results indicate that cellulose may be determined rapidly and with slight error, in presence of moisture (the most significant source of error) and the usual inorg. impurities, by applying the theoretical factor 0.01240 g. of cellulose per ml. of 1.835 N- $\text{K}_2\text{Cr}_2\text{O}_7$  by the heat of dilution method. J. W. D.

**Rapid Accurate Determination of Carbohydrates and Other Substances with the Dichromate Heat-of-dilution Method.** H. F. Launer and Y. Tomimatsu. *Anal. Chem.*, **25**, 1767-1769 (Nov. 1953).

The authors' method (*Food Technol.*, **6**, 59,281 (1952)) in which external heating of the reaction mixture is obviated by utilising the heat of dilution of conc.  $\text{H}_2\text{SO}_4$ , is applicable to starch, sugars, and many types of organic substances; yields  $< 90\%$  with standard deviations  $\geq 0.40$  are recorded. The experimental procedure is described fully. Fifteen substances are cited with which oxidation is incomplete and the method fails. The results are discussed. Presence of an -OH group appears to render a C atom quant. oxidisable; the effect of amino N, itself quant. nonoxidisable, varies greatly. In general organic substances whose C atoms are all individually attached to O atoms, or aromatics, may be successfully determined. Substances which are reproducibly oxidised to  $\text{CO}_2$  plus a fairly stable intermediate such as  $\text{CH}_3\text{COOH}$  may be estimated by taking this product into account theoretically, or determined by standardising the reagent against the pure substance. The method is also applicable to binary mixtures. J. W. D.

Reaction between Chlorous Acid and Glucose—Quantitative Stoichiometry and Evaluation of Reagent Composition (III p. 246).

Influence of the Method of Dyeing on the Wearing Properties of Wool (VIII p. 256).

Preparation of Microbiologically Resistant Wool by means of Chemical Modification. II—Paper-chromatographic Investigation (X p. 257).

Reaction between certain Acid Dyes and Size Precipitate (XI p. 258).

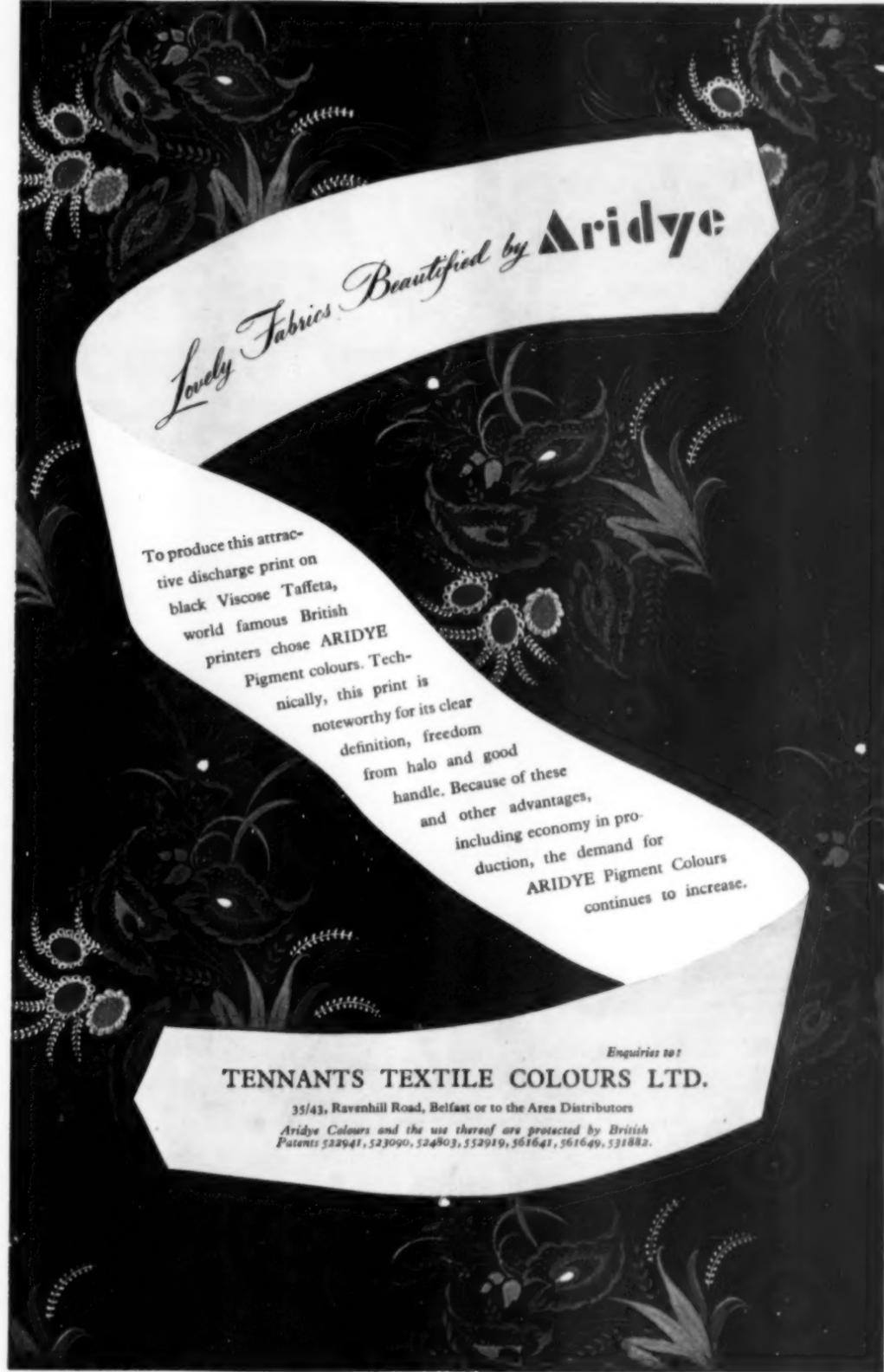
Chromatographic Fractionation of Wood Carbohydrates (XI p. 258).

## XV—MISCELLANEOUS

**Aggregation Tendencies of Acid Wool Dyes in Saline Solutions.** K. N. Davies and J. Whetstone. *J.C.S.*, 865-866 (March 1954).

Previous generalisations as to the nature of the crystal-habit modification processes of inorganic salts with dyes (cf. Whetstone, *Nature*, **168**, 663 (1951)) were derived on the tacit assumption on the basis of results by Speakman and Clegg (*J.S.D.C.*, **50**, 348 (1934)) that, in view of the relatively high degree of sulphonation of most crystal-habit-modifying dyes, single dye molecules rather than dye micelles are the active agents in the modifications. Evidence supporting this assumption is now presented from a direct study of the aggregation tendencies of a number of suitable acid wool dyes, e.g. Acid Magenta, in ammonium nitrate solutions of various concn.

H. H. H.



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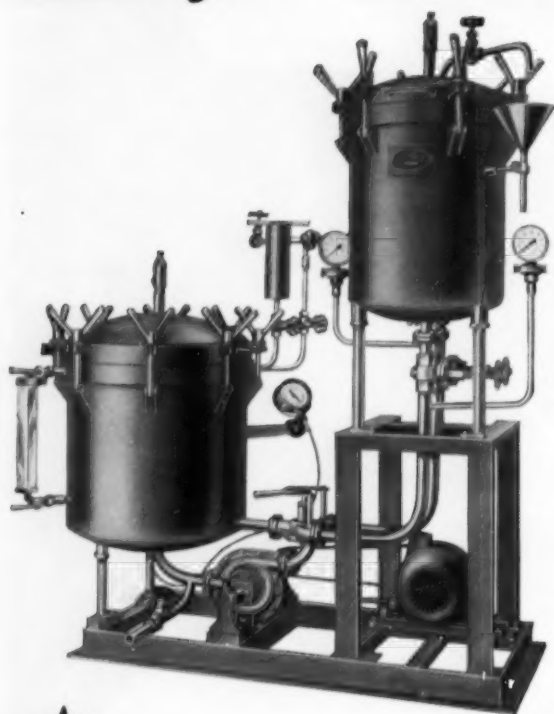
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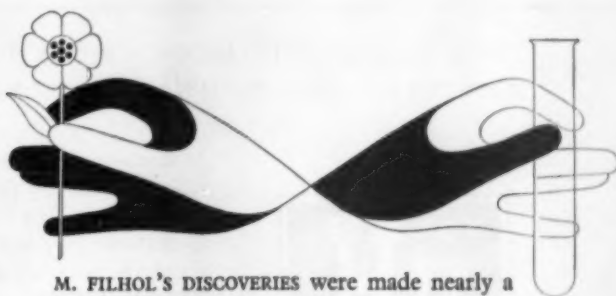
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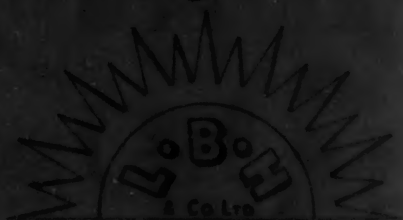
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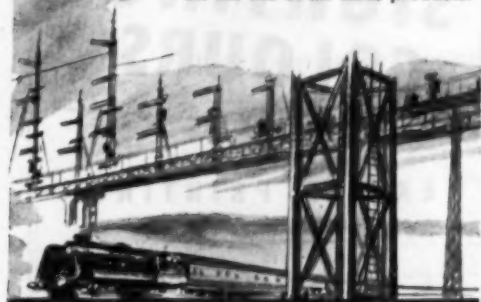


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